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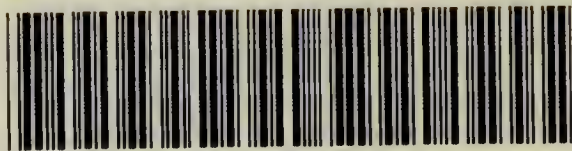
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QUALITATIVE  
CHEMICAL ANALYSIS.



A MANUAL  
OF  
QUALITATIVE  
CHEMICAL ANALYSIS.

BY  
F. BEILSTEIN.

TRANSLATED BY  
WILLIAM RAMSAY, D.Na.Sc.



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1873.

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# P R E F A C E.

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THE translation of the present work has been undertaken with a view to furnish laboratory students with a manual, which should contain the principal methods of Qualitative Chemical Analysis. It is well known and extensively used in Germany, and the name of its author cannot fail to be a guarantee of its excellence. The translator has taken the liberty to supply a few omissions; such, for example, as the tests for aluminium, zinc, etc., by means of cobaltic nitrate, before the blowpipe.

Objections may be raised to the indiscriminate use of symbols to express indefinite quantities of reagents; it is, however, believed that this will familiarise the student with their formulæ; and if he bear in mind that a formula really expresses, not an undetermined amount, but a molecule of the substance, confusion will be avoided.

A full index has also been added, which will supply a want greatly felt by those who are in the habit of using the German edition.

W. R.

GLASGOW, *April, 1873.*



# ATOMIC WEIGHTS.

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## I. MONOVALENT ELEMENTS.

H = 1	K = 39·1
Cl = 35·5	Na = 23
Br = 80	Li = 7
I = 128	Ag = 108
Fl = 19	

## II. DIVALENT ELEMENTS.

O = 16	Ba = 137
S = 32	Sr = 87·5
Hg = 200	Ca = 40
Cu = 63·4	Mg = 24
Cd = 112	Zn = 65

## III. TRIVALENT ELEMENTS.

N = 14	Al = 27·5
P = 31	Cr = 52·5
As = 75	Ur = 118·8
Sb = 122	Fe = 56
Bi = 208	Co = 60
Au = 196	Ni = 58
B = 11	Mn = 55

## IV. TETRAVALENT ELEMENTS.

C = 12	Sn = 118
Si = 28	Pt = 197·4
Tl = 50	Pb = 207

## V. HEXAVALENT ELEMENTS.

Mo = 92	Wo = 184
V = 137	



# CHEMICAL ANALYSIS.

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## EXAMPLES FOR PRACTICE IN ANALYSIS.

### I. SODIC CHLORIDE, NaCl.

*(Common Salt.)*

1. If sodic chloride be heated in a glass tube\* sealed at one end, it decrepitates.

2. A small quantity of sodic chloride when heated in the outer flame of a Bunsen burner, or in the oxidising flame of the blowpipe on the end of a platinum wire, colours it yellow. If a crystal of potassic dichromate, or a slip of paper, tinted with mercuric iodide, be seen in this light, it appears white.

Sodic chloride is easily soluble in water. Its solution is neutral to test paper.

3. Argentic nitrate ( $\text{AgNO}_3$ ), when added to this solution, produces a white curdy precipitate ( $\text{AgCl}$ ), soluble in  $\text{NH}_3$ , but not in  $\text{HNO}_3$ ; so that if the ammoniacal solution be acidified with  $\text{HNO}_3$ ,  $\text{AgCl}$  is again precipitated.

4. Mercurous nitrate  $\text{Hg}_2(\text{NO}_3)_2$  gives a white precipitate of calomel ( $\text{Hg}_2\text{Cl}_2$ ), insoluble in  $\text{HNO}_3$ , which turns black on addition of  $\text{NH}_3$ . ( $\text{Hg}_2\text{Cl}_2 + 2\text{NH}_3 = \text{N}_2\text{Hg}_2\text{H}_6\text{Cl}_2$ .)

\* Before each operation the test tube must be thoroughly washed with distilled water. If solid substances are to be heated, the tube must be carefully dried. This may be easily done by heating the tube over a flame, while the air is sucked out by a glass tube

Plumbic acetate ( $\text{Pb}[\text{C}_2\text{H}_3\text{O}_2]_2$ ); white crystalline precipitate, insoluble in  $\text{HNO}_3$ . Soluble in a large quantity of boiling water, from which long needles of  $\text{PbCl}_2$  are deposited on cooling.

## II. SODIC CARBONATE, $\text{Na}_2\text{CO}_3 + 10\text{H}_2\text{O}$ .

1. On heating in a test tube, water is driven off.
2.  $\text{Na}_2\text{CO}_3$  colours the flame yellow (*vide* I., 2).
3. It is soluble in water. The solution has an alkaline reaction, and effervesces on addition of  $\text{HCl}$ .

## III. SODIC SULPHATE, $\text{Na}_2\text{SO}_4 + 10\text{H}_2\text{O}$ .

1. When heated in a test tube, it parts with its water of crystallization.

2.  $\text{Na}_2\text{SO}_4$  imparts a yellow colour to the flame.

3. If  $\text{Na}_2\text{SO}_4$  be heated on charcoal, in the reducing flame of the blowpipe,  $\text{Na}_2\text{S}$  is formed. The melted mass is detached from the charcoal, placed on a polished silver coin, and moistened with a few drops of water. Where the  $\text{Na}_2\text{S}$  was in contact with the silver, a black stain is left ( $\text{Ag}_2\text{S}$ ).

4.  $\text{BaCl}_2$  added to the neutral aqueous solution, produces a white precipitate of  $\text{BaSO}_4$ , insoluble in acids.

5.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ; white precipitate ( $\text{PbSO}_4$ ), insoluble in acids. On addition of  $\text{H}_2\text{S}$ , the precipitate turns black.  
 $\text{PbSO}_4 + \text{H}_2\text{S} = \text{PbS} + \text{H}_2\text{SO}_4$ .

## IV. POTASSIC NITRATE, $\text{KNO}_3$ .

(*Saltpetre.*)

1. Potassic nitrate melts when heated in a glass tube.
2. When heated on a piece of charcoal before the blowpipe, it deflagrates.
3.  $\text{KNO}_3$  colours the flame violet. If the flame be seen through a glass prism filled with a solution of indigo, it

appears dark red, even when seen through the thickest part of the prism.

4. **Hydric-sodic-tartrate** ( $\text{NaH}_2\text{C}_4\text{H}_4\text{O}_6$ ) when added to a saturated\* solution of potassic nitrate, produces a white precipitate of **hydric-potassic-tartrate** ( $\text{KHC}_4\text{H}_4\text{O}_6$ ). In dilute solutions this precipitate does not form at once, but only after standing for some time. If the inside of the test tube be rubbed with a glass rod, the precipitation takes place more readily.

5. **Platinic chloride** ( $\text{PtCl}_4$ ); yellow precipitate ( $\text{PtCl}_4 \cdot 2\text{KCl}$ ).— $[3\text{PtCl}_4 + 4\text{KNO}_3 = 2(\text{PtCl}_4 \cdot 2\text{KCl}) + \text{Pt}(\text{NO}_3)_4]$ .

6. A portion of the solution is mixed with an equal volume of concentrated sulphuric acid, and on the mixture cooling, a solution of ferrous sulphate is added carefully; at the point of contact of the two fluids, a brown ring is formed ( $2\text{FeSO}_4\text{NO}$ ).— $[6\text{FeSO}_4 + 2\text{HNO}_3 + 3\text{H}_2\text{SO}_4 = 2\text{NO} + 3\text{Fe}_2(\text{SO}_4)_3 + 4\text{H}_2\text{O}]$ .

7. If a solution of  $\text{KNO}_3$  be treated with hydrochloric acid, to which a few drops of indigo solution have been added, and the fluid boiled, the blue colour of the indigo disappears.  $[3\text{C}_8\text{H}_5\text{NO}(\textit{indigo}) + 2\text{HNO}_3 = 3\text{C}_8\text{H}_5\text{NO}_2(\textit{isatin, reddish-yellow}) + 2\text{NO} + \text{H}_2\text{O}]$

8. When dry **potassic nitrate** is heated with copper foil and concentrated  $\text{H}_2\text{SO}_4$ , brownish red vapours are evolved ( $\text{NO}_2$ ).

## V. AMMONIC CHLORIDE, $\text{NH}_4\text{Cl}$ .

1. **Ammonic chloride**, when heated in a tube, sublimes without previously melting.

2. In a concentrated aqueous solution  $\text{PtCl}_4$  produces a yellow crystalline precipitate ( $\text{PtCl}_4 \cdot 2\text{NH}_4\text{Cl}$ ).

3.  $\text{AgNO}_3$ ; white precipitate of  $\text{AgCl}$  (*vide* I. 3).

4.  $\text{NaHC}_4\text{H}_4\text{O}_6$  gives a white crystalline precipitate

\* A saturated solution is prepared by boiling the substance with as small a quantity of the solvent as possible, and filtering off the solution from the undissolved residue.

$(\text{NH}_4\text{HC}_4\text{H}_4\text{O}_6)$ , soluble in a large quantity of  $\text{H}_2\text{O}$ , and in  $\text{NH}_3$ .

5. If the solution of ammoniac chloride be mixed with a concentrated solution of  $\text{NaOH}$  in a basin,  $\text{NH}_3$  escapes, which may be recognised by its smell. The vapour colours red litmus paper blue; and if a rod, moistened with acetic acid, or with dilute  $\text{HCl}$ , be held near the escaping vapour, a white cloud is formed.

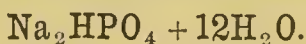
## VI. SODIC NITRATE, $\text{NaNO}_3$ .

1.  $\text{NaNO}_3$  detonates when heated on charcoal. The residue contains sodic carbonate, which, if brought into contact with moist red litmus paper, changes the red colour to blue. The fused mass, treated with  $\text{HCl}$ , evolves  $\text{CO}_2$ .

2. For reactions of  $\text{Na}$ , see I. 2.

3. For reactions of  $\text{HNO}_3$ , see IV. 6.

## VII. HYDRIC-DISODIC PHOSPHATE,



1. When heated in a tube, it gives off water. The aqueous solution has an alkaline reaction.

2.  $\text{AgNO}_3$  produces a yellow precipitate ( $\text{Ag}_3\text{PO}_4$ ), soluble in  $\text{NH}_3$ , and in  $\text{HNO}_3$ .

3.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ; white precipitate ( $\text{Pb}_3[\text{PO}_4]_2$ ) soluble in  $\text{HNO}_3$ .

4.  $\text{CaCl}_2$ , or  $\text{BaCl}_2$ ; white precipitate ( $\text{CaHPO}_4$ , or  $\text{BaHPO}_4$ ), soluble in  $\text{HCl}$ . Ammonia added to the solution in  $\text{HCl}$ , reproduces the precipitate. ( $\text{Ca}_3[\text{PO}_4]_2$  or  $\text{Ba}_3[\text{PO}_4]_2$ ) — ( $3\text{CaHPO}_4 + 3\text{NH}_3 = \text{Ca}_3[\text{PO}_4]_2 + (\text{NH}_4)_3(\text{PO}_4)$ ).

5.  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$  and  $\text{MgSO}_4$  are added to the solution of  $\text{Na}_2\text{HPO}_4$ . A white precipitate ( $\text{Mg}[\text{NH}_4]\text{PO}_4$ ) is thrown down.



VIII. SODIC DIBORATE,  $\text{Na}_2\text{B}_4\text{O}_7 + 10\text{H}_2\text{O}$ .

(Borax.)

1. When heated in a tube, sodic diborate bubbles up, and gives off water.

2. When heated on charcoal, the same takes place; finally, however, it melts, and forms a colourless transparent bead. The concentrated solution has a slightly alkaline reaction.

3. If a hot saturated aqueous solution be treated with concentrated  $\text{H}_2\text{SO}_4$ , glancing crystals ( $\text{B}[\text{OH}]_3$ ), deposit from the liquid on cooling.

4.  $\text{CaCl}_2$ , or  $\text{BaCl}_2$ , when added to a concentrated solution of borax, give white precipitates, soluble in excess of water.

5. Turmeric paper, after having been dipped in a solution of sodic diborate previously acidified with  $\text{HCl}$ , turns brown on drying.

6. Some sodic diborate is placed in a basin, moistened with  $\text{H}_2\text{SO}_4$ , and alcohol added; on applying light, the alcohol burns with a green flame, which may be intensified by stirring the mixture. The flame is rendered more distinct, if, after allowing the alcohol to burn for some time, it be blown out, and again lighted.

For reactions of sodium, *vide* I. 2.

IX. MAGNESIC SULPHATE,  $\text{MgSO}_4 + 7\text{H}_2\text{O}$ .

1. On heating in a tube, water is given off.

2. On heating with  $\text{Na}_2\text{CO}_3$  on charcoal, in the reduction flame,  $\text{Na}_2\text{S}$  is formed (*vide* III. 3).

In aqueous solution,

3.  $\text{NaOH}$ , or  $\text{NH}_3$  gives a white precipitate ( $\text{Mg}[\text{OH}]_2$ ), soluble in  $\text{NH}_4\text{Cl}$ .

4.  $\text{Na}_2\text{CO}_3$ ; white precipitate, soluble in  $\text{NH}_4\text{Cl}$ .

5. Ammonic carbonate ( $[\text{NH}_4]_2\text{CO}_3$ ) gives no precipitate; but on addition of  $\text{Na}_2\text{HPO}_4$ ,  $(\text{NH}_4)\text{MgPO}_4$

separates out (*vide* VII. 5).— $(\text{MgSO}_4 + (\text{NH}_4)_2\text{CO}_3 + \text{Na}_2\text{HPO}_4 = \text{Mg}(\text{NH}_4)\text{PO}_4 + \text{Na}_2\text{SO}_4 + \text{NH}_4\text{HCO}_3)$ .  
For reactions of  $\text{H}_2\text{SO}_4$ , see III. 4.

## X. CALCIC CARBONATE, $\text{CaCO}_3$ .

(*Marble, Chalk.*)

1.  $\text{CaCO}_3$  is not altered by being heated in a tube.
2. When heated on charcoal,  $\text{CO}_2$  is evolved. The residue possesses an alkaline reaction (VI. 1).
3.  $\text{CaCO}_3$  is insoluble in  $\text{H}_2\text{O}$ , but dissolves in  $\text{HCl}$  with effervescence. If the gas which is evolved be led into clear lime-water, turbidity is immediately produced, or if a drop of baryta-water be held close over the effervescing fluid, it becomes turbid ( $\text{BaCO}_3$ ).
4. Prepare a neutral solution of  $\text{CaCl}_2$ , by treating chalk or marble with a quantity of  $\text{HCl}$ , insufficient to dissolve it. After the evolution of gas has ceased, the solution is heated to boiling, and filtered off from the undissolved marble. If all dissolves, more  $\text{CaCO}_3$  must be added.

From this solution,

5.  $\text{NaOH}$  precipitates  $\text{Ca}(\text{OH})_2$ , soluble in a large excess of water.
6.  $\text{NH}_3$  gives no precipitate, but  $(\text{NH}_4)_2\text{CO}_3$ , or  $\text{Na}_2\text{CO}_3$ , throws down a white precipitate of  $\text{CaCO}_3$ .
7.  $\text{Na}_2\text{HPO}_4$ ; white precipitate ( $\text{CaHPO}_4$ , *vide* VII. 4), soluble in  $\text{HC}_2\text{H}_3\text{O}_2$ , and in all inorganic acids.
8. Ammonic oxalate ( $[\text{NH}_4]_2\text{C}_2\text{O}_4$ ); white precipitate ( $\text{CaC}_2\text{O}_4$ ), insoluble in  $\text{HC}_2\text{H}_3\text{O}_2$ , soluble in  $\text{HCl}$ , or in  $\text{HNO}_3$ . It is precipitated again from these solutions by  $\text{HNO}_3$ .

9.  $\text{H}_2\text{SO}_4$  gives a white precipitate ( $\text{CaSO}_4$ ) only in concentrated solutions, soluble in a large quantity of water, and also in  $\text{HCl}$ . It is precipitated from these solutions on addition of alcohol.

### XI. BARIC CHLORIDE, $\text{BaCl}_2 + 2\text{H}_2\text{O}$ .

1. If heated in a tube,  $\text{BaCl}_2$  gives off its water of crystallization.

2. It fuses when heated on charcoal. The residue has an alkaline reaction.

3.  $\text{BaCl}_2$  colours the flame greenish-yellow.

4.  $\text{NaOH}$ , added to a concentrated aqueous solution, produces a white precipitate of  $\text{Ba}(\text{OH})_2$ , soluble in boiling water.

5.  $\text{NH}_3$  gives no precipitate, but  $(\text{NH}_4)_2\text{CO}_3$  gives a white precipitate of  $\text{BaCO}_3$ .

6.  $\text{H}_2\text{SO}_4$ ; white precipitate ( $\text{BaSO}_4$ ), insoluble in acids (*vide* III. 4).

7. A solution of  $\text{CaSO}_4$  produces turbidity in solutions of salts of barium, owing to the great insolubility of the resulting  $\text{BaSO}_4$ .

8.  $\text{Na}_2\text{HPO}_4$ ; white precipitate ( $\text{BaHPO}_4$ ), soluble in  $\text{HCl}$  (*vide* VII. 4).

9. Hydro-fluo-silicic acid ( $\text{SiFl}_4 \cdot 2\text{HFl}$ ); white precipitate ( $\text{SiFl}_4 \cdot \text{BaFl}_2$ ).

10.  $\text{K}_2\text{Cr}_2\text{O}_7$ ; yellow precipitate ( $\text{BaCrO}_4$ ), soluble in  $\text{HCl}$ , but on addition of  $\text{NH}_3$  is again precipitated.—  
 $(\text{BaCl}_2 + \text{K}_2\text{Cr}_2\text{O}_7 = \text{BaCrO}_4 + \text{CrO}_3 + 2\text{KCl})$ .

For reactions of  $\text{Cl}$  *vide* I. 3.

### XII. STRONTIC CHLORIDE, $\text{SrCl}_2 + 6\text{H}_2\text{O}$ .

1. When heated in a glass tube  $\text{SrCl}_2$  gives off water.

2. It behaves like  $\text{BaCl}_2$  (XI. 2), when heated before the blowpipe.

3. It imparts to the flame a brilliant crimson colour.

4.  $\text{NaOH}$ ,  $\text{NH}_3$ ,  $\text{Na}_2\text{CO}_3$  (or  $[\text{NH}_4]_2\text{CO}_3$ ),  $\text{Na}_2\text{HPO}_4$ , when added to an aqueous solution of  $\text{SrCl}_2$ , produce reactions analogous to those of  $\text{BaCl}_2$  (XI. 4, 5, 8).

5.  $\text{H}_2\text{SO}_4$ ; white precipitate ( $\text{SrSO}_4$ ), insoluble in acids.

6.  $\text{CaSO}_4$  produces a slight turbidity after standing for some time.

7.  $\text{SiF}_4$ ,  $2\text{HF}$ , and  $\text{K}_2\text{Cr}_2\text{O}_7$ , give no precipitates.

### XIII. CALCIC SULPHATE, $\text{CaSO}_4 + 2\text{H}_2\text{O}$ .

(*Gypsum.*)

1. When heated in a tube, water is given off.

2. When heated before the blowpipe with sodic carbonate, it is reduced to sulphide.

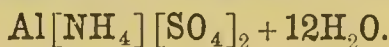
It dissolves with difficulty in  $\text{H}_2\text{O}$ , but more easily in  $\text{HNO}_3$ .

3.  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ , when added to the aqueous solution produces a white precipitate  $\text{CaC}_2\text{O}_4$ .

4.  $\text{BaCl}_2$ ; white precipitate ( $\text{BaSO}_4$ ).

5. Alcohol added to the aqueous solution produces a white precipitate ( $\text{CaSO}_4$ ), owing to its not being so soluble in a mixture of alcohol and water (X. 9).

### XIV. AMMONIC ALUMINIC SULPHATE,



(*Ammonia Alum.*)

1. When heated in a glass tube, water is evolved.

2. It is converted into sulphides when heated before the blowpipe with  $\text{Na}_2\text{CO}_3$ .

3. Ammonia when added to the aqueous solution produces a white precipitate ( $\text{Al}[\text{OH}]_3$ ), insoluble in excess of the precipitant.

4.  $\text{NaOH}$ ; white precipitate ( $\text{Al}[\text{OH}]_3$ ), soluble in excess, but again precipitable by  $\text{NH}_4\text{Cl}$ .

5.  $\text{Na}_2\text{CO}_3$ ; white precipitate ( $\text{Al}[\text{OH}]_3$ ), with evolution of  $\text{CO}_2$ .  $(3\text{Na}_2\text{CO}_3 + 2(\text{NH}_4)\text{Al}(\text{SO}_4)_2 + 3\text{H}_2\text{O} = 2\text{Al}(\text{OH})_3 + 3\text{CO}_2 + 3\text{Na}_2\text{SO}_4 + [\text{NH}_4]_2\text{SO}_4)$ .

6.  $(\text{NH}_4)_2\text{S}$ ; white precipitate ( $\text{Al}[\text{OH}]_3$ ), with evolu-

tion of  $H_2S$ .  $(3[NH_4]_2S + 2[NH_4]Al[SO_4]_2 + 6H_2O = 2Al(OH)_3 + 3H_2S + 4[NH_4]_2SO_4)$ .

Reactions for  $H_2SO_4$ , *vide* III. 4. For those of ammonia, *vide* V. 5.

### XV. POTASSIC DICHROMATE, $K_2Cr_2O_7$ .

1. When heated in a glass tube, it fuses, forming a dark red fluid.

2. It deflagrates slightly when heated on charcoal. The residue consists of  $Cr_2O_3$  and  $K_2CO_3$ .

3. Before the blowpipe with microcosmic salt or borax, it gives a green bead, both in the reducing and oxidising flames.

4.  $(NH_4)_2S$ , added to the orange-coloured aqueous solution, gives a brown precipitate ( $Cr_2[CrO_4]_3$ ), accompanied by the separation of sulphur. The solution contains yellow  $K_2CrO_4$ . ( $5K_2Cr_2O_7 + 3(NH_4)_2S = Cr_2(CrO_4)_3 + S_3 + 5K_2CrO_4 + 6NH_3 + 3H_2O$ ). If the solution, after being saturated with  $(NH_4)_2S$ , be heated to boiling, green  $Cr[OH]_3$  is precipitated, besides more sulphur ( $K_2Cr_2O_7 + 3[NH_4]_2S + H_2O = 2Cr[OH]_3 + S_3 + 2KOH + 6NH_3$ ).

5.  $(PbCr_2H_3O_2)_2$ ; yellow precipitate ( $PbCrO_4$ ), insoluble in dilute  $HNO_3$ , but dissolving readily in  $NaOH$ .

6.  $Hg_2(NO_3)_2$ ; bright red precipitate ( $Hg_2CrO_4$ ).

7.  $AgNO_3$ ; brownish red precipitate ( $Ag_2CrO_4$ ).

8. The aqueous solution of  $SO_2$ , reduces potassic dichromate to the state of green chromic salt.

9. When boiled with concentrated  $HCl$ , chlorine is given off, and a green solution of  $CrCl_3$  remains behind. The reaction is more easily obtained on addition of some drops of alcohol ( $K_2Cr_2O_7 + 8HCl + 3C_2H_6O = 2CrCl_3 + 2KCl + 3C_2H_4O$  (aldehyde) +  $7H_2O$ ).

10.  $NaOH$ , added to this solution, produces a green precipitate ( $Cr[OH]_3$ ), which is soluble in excess of the precipitant, but is re-precipitated on boiling.

11.  $NH_3$ ; a precipitate of a greyish-blue colour,

slightly soluble in excess. The solution has a reddish colour.

12.  $(\text{NH}_4)_2\text{S}$ ; precipitate of  $\text{Cr}[\text{OH}]_3$ .

13.  $\text{Na}_2\text{CO}_3$ ; green precipitate of a basic salt.

## XVI. IRON, Fe.

1. A thin piece of iron wire burns when held in the blowpipe flame, forming  $\text{Fe}_3\text{O}_4$ .

2. Iron dissolves in warm  $\text{HCl}$ , with evolution of  $\text{H}$ , and deposits a residue of insoluble carbon. The solution should contain excess of iron, and should be boiled till the evolution of  $\text{H}$  ceases.

3. In this solution of  $\text{FeCl}_2$  (Ferrous chloride)  $\text{NaOH}$ , or  $\text{NH}_3$ , produces a greenish-white precipitate ( $\text{Fe}[\text{OH}]_2$ ), which turns first green, and finally reddish-brown, when exposed to the air, forming  $\text{Fe}(\text{OH})_3$ .

4.  $\text{Na}_2\text{CO}_3$ ; white precipitate, turning rapidly brown by absorption of oxygen.

5.  $(\text{NH}_4)_2\text{S}$ ; black precipitate ( $\text{FeS}$ ), soluble in  $\text{HCl}$ , with evolution of  $\text{H}_2\text{S}$ .

Boil the solution of  $\text{FeCl}_2$  with a few drops of  $\text{HNO}_3$ . It rapidly assumes a yellow colour, and contains ferric salts; at the same time  $\text{N}_2\text{O}_2$  is given off. ( $3\text{FeCl}_2 + 4\text{HNO}_3 = 2\text{FeCl}_3 + \text{Fe}(\text{NO}_3)_3 + \text{NO} + 2\text{H}_2\text{O}$ .)

6.  $\text{NaOH}$ ,  $\text{NH}_3$ , or  $\text{Na}_2\text{CO}_3$  added to this solution give reddish-brown precipitates ( $\text{Fe}[\text{OH}]_3$ ).

7.  $\text{H}_2\text{S}$ ; pure white precipitate of  $\text{S}$ . The solution contains iron in the dyad state.

8.  $(\text{NH}_4)_2\text{S}$ ; black precipitate of  $\text{FeS}$  and  $\text{S}$ .

9. Potassic-ferrocyanide ( $4\text{KCN} \cdot \text{Fe}[\text{CN}]_2$ ); dark blue precipitate (Prussian blue —  $\text{Fe}_7[\text{CN}]_{13} = 4\text{FeCN}_3 \cdot 3\text{Fe}[\text{CN}]_2$ ).

10. Potassic sulphocyanide ( $\text{KCNS}$ ); blood-red colouration ( $\text{Fe}[\text{CNS}]_3$ ).

11.  $\text{BaCO}_3$  suspended in water gives a brown precipitate of  $\text{Fe}(\text{OH})_3$ , with evolution of  $\text{CO}_2$ .

12. If a portion of the precipitates, obtained as directed

in XVI. 3-6, be heated before the blowpipe in the reducing flame, with a bead of microcosmic salt, a green bead is produced, which turns red when held in the oxidising part of the flame. When cold the colour of the bead will have partially or totally disappeared.

## XVII. MANGANIC DIOXIDE, $MnO_2$ .

1. When heated strongly, in a glass tube, it yields up a portion of its oxygen. A glowing splinter of wood, when inserted into the tube, commences to burn with a bright flame.

2.  $MnO_2$  is insoluble in  $H_2O$ , in  $H_2SO_4$ , and in  $HNO_3$ ; but dissolves in  $HCl$ , with evolution of  $Cl$ .

3. Boil some  $MnO_2$  with concentrated  $HCl$ , till the chlorine is no longer given off, and filter off from the excess of  $MnO_2$ . Add  $Na_2CO_3$  till a precipitate is produced, which does not disappear on shaking the liquid. Add a few drops of  $H_2C_2H_3O_2$  to dissolve the precipitate, and then an excess of  $NaC_2H_3O_2$ , and boil. The resulting precipitate of basic ferric acetate is filtered off, dissolved in  $HCl$ , and the presence of iron proved according to the directions in XVI. 9, 10.

In the filtrate, which contains the manganese,

4.  $NaOH$  or  $NH_3$  gives a white precipitate of  $Mn(OH)_2$ , rapidly turning brown in the air ( $Mn_2O_3$ ). In presence of  $NH_4Cl$ ,  $NH_3$  produces no precipitate.

5.  $Na_2CO_3$  or  $(NH_4)_2CO_3$ ; white precipitate ( $MnCO_3$ ).

6.  $(NH_4)_2S$ ; flesh-coloured precipitate of  $MnS$ , soluble in  $HCl$ .

7.  $MnO_2$ , boiled with  $Pb_3O_4$  and  $HNO_3$ , is oxidised to  $HMnO_4$  (permanganic acid), the solution of which has a deep red colour.

8. When heated with a bead of microcosmic salt before the blowpipe,  $MnO_2$  gives an amethyst-coloured bead, which becomes colourless in the reducing flame.

9. If  $MnO_2$  and  $Na_2CO_3$ , with the addition of a small

quantity of  $\text{KNO}_3$ , be fused on a piece of platinum foil, a greenish-blue mass results (Sodic manganate  $\text{Na}_2\text{MnO}_4$ ).

### XVIII. ZINC, Zn.

1. When heated on charcoal, it burns with a white flame, giving a white smoke ( $\text{ZnO}$ ), and leaving a deposit on the charcoal, which is yellow when hot, and grows white on cooling, and which is not volatile.

2. Zn is soluble in  $\text{HCl}$ , with evolution of H. Boil excess of Zn with  $\text{HCl}$  till the effervescence ceases, and filter off the colourless solution of  $\text{ZnCl}_2$ .

3.  $\text{NaOH}$ , or  $\text{NH}_3$ , added to this solution give a white precipitate ( $\text{Zn}[\text{OH}]_2$ ) soluble in excess of the precipitant.

4.  $\text{Na}_2\text{CO}_3$ ; white precipitate of a basic salt. Carbonic anhydride is evolved.

5.  $\text{H}_2\text{S}$ ; white precipitate ( $\text{ZnS}$ ), soluble in  $\text{HCl}$ . If the solution of  $\text{ZnCl}_2$  contain much free  $\text{HCl}$ ,  $\text{H}_2\text{S}$  fails to give a precipitate.

6.  $(\text{NH}_4)_2\text{S}$ ; white precipitate ( $\text{ZnS}$ ), insoluble in  $\text{HC}_2\text{H}_3\text{O}_2$ .

### XIX. NICKEL SULPHATE, $\text{NiSO}_4 + 7\text{H}_2\text{O}$ .

1.  $\text{NaOH}$ , added to the aqueous solution, produces a green precipitate of  $\text{Ni}(\text{OH})_2$ .

2.  $\text{NH}_3$ ;  $\text{Ni}(\text{OH})_2$ , soluble in excess of the precipitant.

3.  $\text{Na}_2\text{CO}_3$ ; green precipitate of a basic salt ( $2\text{NiCO}_3 \cdot 3\text{Ni}[\text{OH}]_2$ ).

4.  $(\text{NH}_4)_2\text{CO}_3$ ; green precipitate, soluble in excess.

5.  $(\text{NH}_4)_2\text{S}$ ; black precipitate ( $\text{NiS}$ ), insoluble in  $\text{HCl}$ . If a portion of this precipitate be heated with a bead of microcosmic salt, in the blowpipe flame, the bead assumes a colour, varying, when hot, from red to reddish-brown, and when cold from yellow to orange.



XX. COBALTIC NITRATE,  $\text{Co}(\text{NO}_3)_2 + 5\text{H}_2\text{O}$ .

1.  $\text{NaOH}$  precipitates a blue-coloured basic salt from the aqueous solution of cobaltic nitrate; on boiling, the precipitate changes to a dirty red colour ( $\text{Co}[\text{OH}]_2$ ).

2.  $\text{NH}_3$ ; blue precipitate of a basic salt, soluble in excess of the precipitant to a red liquid.

3.  $(\text{NH}_4)_2\text{S}_2$ ; black precipitate ( $\text{CoS}$ ), insoluble in  $\text{HCl}$ . Some of the precipitate, investigated before the blowpipe gives, in either flame, a blue bead.

4. Potassic nitrite ( $\text{KNO}_2$ ), added to a solution which has been acidified with  $\text{HC}_2\text{H}_3\text{O}_2$ , produces a yellow precipitate after some time ( $\text{Co}[\text{OH}]\text{K}_3[\text{NO}_2]_5 \cdot (\text{Co}[\text{NO}_3]_2 + 6\text{KNO}_2 + \text{HC}_2\text{H}_3\text{O}_2 = \text{Co}[\text{OH}]\text{K}_3[\text{NO}_2]_5 + 2\text{KNO}_3 + \text{KC}_2\text{H}_3\text{O}_2 + \text{NO})$ ).

XXI. LEAD,  $\text{Pb}$ .

1. Lead, when heated on charcoal before the blowpipe, melts, and coats the charcoal with a yellow deposit ( $\text{PbO}$ ).

2. Lead is insoluble in  $\text{HCl}$ , and in  $\text{H}_2\text{SO}_4$ , but dissolves in  $\text{HNO}_3$ , with evolution of  $\text{NO}$ . ( $3\text{Pb} + 8\text{HNO}_3 = 3\text{Pb}[\text{NO}_3]_2 + 2\text{NO} + 4\text{H}_2\text{O}$ ).

3. From neutral solutions of lead, zinc precipitates metallic lead in a crystalline state.

4.  $\text{H}_2\text{S}$ ; black  $\text{PbS}$ , insoluble in acids. When boiled with concentrated nitric acid, it is resolved into  $\text{PbSO}_4$ ; white, and insoluble in acids.

5.  $\text{NaOH}$ ; white precipitate ( $\text{Pb}[\text{OH}]_2$ ), soluble in a large excess of the precipitant.

6.  $\text{NH}_3$ ; white precipitate of a basic salt, soluble in excess.

7.  $\text{Na}_2\text{CO}_3$ ; white  $\text{PbCO}_3$ .

8.  $\text{H}_2\text{SO}_4$ ; white  $\text{PbSO}_4$ , insoluble in acids, but easily soluble in ammoniacal solution of  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ . On addition of  $(\text{NH}_4)_2\text{S}$ ,  $\text{PbSO}_4$  turns into  $\text{PbS}$ .

9.  $\text{HCl}$ ; white  $\text{PbCl}_2$ , soluble in a large quantity of boiling water (*vide* I. 5).

10.  $\text{K}_2\text{Cr}_2\text{O}_7$ ; yellow precipitate of  $\text{PbCrO}_4$  (*vide* XV. 5).

11.  $(\text{NH}_4)_2\text{S}$ ; black precipitate ( $\text{PbS}$ ).

## XXII. BISMUTH, Bi.

1. When heated in a glass tube, metallic bismuth fuses without volatilising.

2. If heated on charcoal before the blowpipe it fuses, giving a yellow incrustation ( $\text{Bi}_2\text{O}_3$ ) and a brittle metallic globule.

Bismuth is soluble in concentrated  $\text{HNO}_3$  with evolution of  $\text{NO}$ .

3.  $\text{H}_2\text{O}$ , added to a solution of Bi in  $\text{HNO}_3$ , throws down a white, crystalline, basic salt ( $\text{BiONO}_3 + \text{H}_2\text{O}$ ). If much nitric acid be present, the precipitate does not appear till some  $\text{NaCl}$ , or  $\text{HCl}$ , has been added. In this case, *bismuthyl chloride* ( $\text{BiO.Cl}$ ) is formed ( $\text{Bi}[\text{OH}]_2\text{NO}_3 + \text{NaCl} = \text{BiO.Cl} + \text{NaNO}_3 + \text{H}_2\text{O}$ ).

4.  $\text{H}_2\text{S}$ , or  $(\text{NH}_4)_2\text{S}$ ; blackish-brown precipitate of  $\text{Bi}_2\text{S}_3$ , soluble neither in  $(\text{NH}_4)_2\text{S}$ , nor in dilute acids.

5.  $\text{NaOH}$ , or  $\text{NH}_3$ ; white precipitate ( $\text{Bi}[\text{OH}]_3$ ), insoluble in excess.

6.  $\text{K}_2\text{Cr}_2\text{O}_7$ ; yellow precipitate ( $[\text{BiO}]\text{Bi}[\text{CrO}_4]_2$ ), insoluble in  $\text{NaOH}$ , but soluble in  $\text{HNO}_3$ . ( $2\text{Bi}[\text{NO}_3]_3 + \text{K}_2\text{Cr}_2\text{O}_7 + 2\text{H}_2\text{O} = [\text{BiO}]\text{Bi}[\text{CrO}_4]_2 + 2\text{KNO}_3 + 4\text{HNO}_3$ ).

## XXIII. COPPER, Cu.

Copper is soluble in  $\text{HNO}_3$ , with evolution of  $\text{NO}$ .

1.  $\text{Fe}$  (iron wire) precipitates metallic copper from the solution of cupric nitrate ( $\text{Cu}[\text{NO}_3]_2$ ).

2.  $\text{NaOH}$ ; blue precipitate ( $\text{Cu}[\text{OH}]_2$ ), which turns black on being boiled ( $\text{CuO}$ ).

3.  $\text{NH}_3$ ; greenish-blue precipitate of a basic salt,

which dissolves in excess of the precipitant with a deep blue colour ( $\text{Cu}[\text{NO}_3]_2 \cdot 4\text{NH}_3$ ).

4.  $\text{H}_2\text{S}$ ; black precipitate ( $\text{CuS}$ ). Heat a portion of the precipitate, mixed with sodic carbonate, on charcoal before the blowpipe, and reduce the melted mass to powder, by rubbing in an agate mortar with a little water. If the particles of carbon be removed by decantation, small glancing particles of metallic copper are left behind.

5.  $(\text{NH}_4)_2\text{S}$ ; black precipitate ( $\text{CuS}$ ), insoluble in dilute acids in the cold, and almost insoluble in the precipitant. Before the blowpipe the precipitate, when heated on borax in the oxidising flame, gives a green bead, which becomes red and opaque when removed to the reducing flame.

6.  $4\text{KCN} \cdot \text{Fe}(\text{CN}_2)$ ; reddish-brown precipitate ( $2\text{Cu}[\text{CN}]_2 \cdot \text{Fe}[\text{CN}]_2$ ), insoluble in acids.

#### XXIV. CADMIUM, Cd.

1. Cadmium heated before the blowpipe fuses and volatilises, leaving a brown incrustation on the charcoal ( $\text{CdO}$ ).

2. Cadmium dissolves slowly in  $\text{H}_2\text{SO}_4$  and in  $\text{HCl}$ , but easily in  $\text{HNO}_3$  to  $\text{Cd}(\text{NO}_3)_2$ .

3. Metallic  $\text{Zn}$  precipitates from this solution metallic cadmium.

4.  $\text{H}_2\text{S}$ ; yellow  $\text{CdS}$ , insoluble in  $\text{NH}_3$ .

5.  $(\text{NH}_4)_2\text{S}$ ; yellow  $\text{CdS}$ , insoluble in the precipitant.

6.  $\text{NaOH}$ ; white  $\text{Cd}(\text{OH})_2$ , insoluble in excess.

7.  $\text{NH}_3$ ; white  $\text{Cd}(\text{OII})_2$ , easily soluble in excess of  $\text{NH}_3$ .

8.  $\text{Na}_2\text{CO}_3$ ; white  $\text{CdCO}_3$ .

#### XXV. MERCURY, Hg.

1. Mercury dissolves with ease, in *slightly* warm  $\text{HNO}_3$ . The solution may be decanted from the excess of mercury, and consists of  $\text{Hg}_2(\text{NO}_2)_2$ .

2. Cu (copper-foil); metallic mercury.
3. SO<sub>2</sub>; gray, metallic mercury.
4. H<sub>2</sub>S, or (NH<sub>4</sub>)<sub>2</sub>S; black Hg<sub>2</sub>S.
5. NaHO; black Hg<sub>2</sub>O.
6. NH<sub>3</sub>; black amido-compounds (N.Hg<sub>2</sub>H<sub>2</sub>HNO<sub>3</sub>).
7. HCl; white Hg<sub>2</sub>Cl<sub>2</sub> (calomel).
8. KI; greenish-yellow Hg<sub>2</sub>I<sub>2</sub>.

Add to the solution of Hg<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> some concentrated HNO<sub>3</sub>, a few drops of HCl, and boil till the precipitate disappears.

In this solution (Hg[NO<sub>3</sub>]<sub>2</sub>),

9. Cu; metallic mercury.
10. Stannous chloride (SnCl<sub>2</sub>); a white precipitate of Hg<sub>2</sub>Cl<sub>2</sub> is produced; on addition of more SnCl<sub>2</sub>, gray, metallic mercury.
11. NaOH; brown precipitate of a basic salt; on addition of more NaOH, is changed into yellow HgO. Heat a portion of the latter precipitate in a glass tube, when metallic mercury will sublime, and may be formed into a globule by rubbing.
12. NH<sub>3</sub>; white precipitate of an amido-compound.—(N<sub>2</sub>Hg<sub>3</sub>H<sub>2</sub>[NO<sub>3</sub>]<sub>2</sub>.)
13. Na<sub>2</sub>CO<sub>3</sub>; reddish-brown precipitate of a basic salt (HgCO<sub>3</sub>.3HgO).
14. H<sub>2</sub>S, or (NH<sub>4</sub>)<sub>2</sub>S, added in small quantity, produce a white precipitate (2HgS.Hg[NO<sub>3</sub>]<sub>2</sub>), which by addition of more of the precipitant turns to HgS.
15. KI; red precipitate (HgI<sub>2</sub>), soluble in excess of KI.
16. HCl produces no precipitate.

## XXVI. SILVER, Ag.

1. Dissolve a silver coin in HNO<sub>3</sub>, and precipitate the silver as chloride (AgCl) by adding HCl to the solution, which has a blue colour owing to the presence of copper (Cu[NO<sub>3</sub>]<sub>2</sub>). Shake the solution and filter it. The precipitate (AgCl) is well washed with hot water, dried, and fused in a porcelain crucible. As soon as the melted

mass has grown cold, a little water is poured on the top, a piece of zinc laid on it, and a few drops of HCl added. After a few hours the undissolved zinc is removed, the metallic silver boiled with some HCl, and washed with water repeatedly by decantation, then dissolved in  $\text{HNO}_3$ . The solution of  $\text{AgNO}_3$  is evaporated carefully to dryness, and the dry residue dissolved in  $\text{H}_2\text{O}$ . The solution is filtered.

2. **Cu**; metallic Ag. The blue solution contains  $\text{Cu}(\text{NO}_3)_2$ .

3.  $\text{H}_2\text{S}$ , or  $(\text{NH}_4)_2\text{S}$ ; black precipitate ( $\text{Ag}_2\text{S}$ ).

4. **NaOH**; dark brown precipitate ( $\text{Ag}_2\text{O}$ ).

5.  $\text{NH}_3$ ; brown precipitate ( $\text{Ag}_2\text{O}$ ), easily soluble in excess of the precipitant. If free acid be present in the solution of  $\text{AgNO}_3$ ,  $\text{NH}_3$  produces no precipitate.

6.  $\text{Na}_2\text{HPO}_4$ ; yellow precipitate ( $\text{Ag}_3\text{PO}_4$ ), soluble in  $\text{NH}_3$  and in  $\text{HNO}_3$ .

7. **HCl**; white curdy precipitate of  $\text{AgCl}$ , soluble in  $\text{NH}_3$ , insoluble in  $\text{HNO}_3$ . If exposed to the light, it assumes a violet colour.

## XXVII. TIN, Sn.

1. When heated on charcoal, before the blowpipe, it fuses, and when exposed to the outer flame, is oxidised to  $\text{SnO}_2$ .

2. Tin is soluble in concentrated boiling HCl, with evolution of H.

3. **Zn** precipitates crystalline metallic Sn from this solution.

4. **NaOH**; white  $\text{Sn}(\text{OH})_2$ , soluble in excess.

5.  $\text{NH}_3$ ; white  $\text{Sn}(\text{OH})_2$ , insoluble in excess.

6.  $\text{Na}_2\text{CO}_3$ ; white  $\text{Sn}(\text{OH})_2$ , with evolution of  $\text{CO}_2$ .

7.  $\text{H}_2\text{S}$ ; dark brown precipitate ( $\text{SnS}$ ).

8.  $(\text{NH}_4)_2\text{S}$ ; dark brown  $\text{SnS}$ , soluble in yellow ammoniac sulphide (*i.e.*,  $[\text{NH}_4]_2\text{S}$ , in which sulphur is held in solution). HCl added to this solution gives a yellow precipitate of  $\text{SnS}_2$ .

If Sn be heated with  $\text{HNO}_3$ , it forms white insoluble metastannic oxide ( $\text{SnO}_2$ ). As soon as the precipitate has a pure white colour, the acid is poured off, the residue washed with water by means of decantation, boiled with concentrated  $\text{HCl}$ , and water added to the solution, which is then filtered.

9.  $\text{NaOH}$  produces a white precipitate ( $\text{SnO}[\text{OH}]_2$ ), soluble in excess.

10.  $\text{NH}_3$ ; white precipitate of  $\text{SnO}(\text{OH})_2$ , insoluble in excess.

11. Treat a portion of the precipitate as directed in XXIII. 4. White, glancing, malleable, metallic beads are obtained.

12. The solution of Sn is diluted largely with water, and boiled. White precipitate,  $\text{SnO}(\text{OH})_2$ .

### XXVIII. ANTIMONY, Sb.

1. When antimony is heated in a glass tube it fuses, but does not sublime.

2. If heated before the blowpipe, on charcoal, it melts, and gives a white, volatile incrustation ( $\text{Sb}_2\text{O}_3$ ). The fused bead of antimony continues to burn spontaneously, forming white vapours, and becomes covered with needle-shaped crystals of  $\text{Sb}_2\text{O}_3$ .

3. Antimony is insoluble in  $\text{HCl}$ ;  $\text{HNO}_3$  changes it to white, insoluble  $\text{SbO}_2$ ; but in a mixture of these acids (three volumes of the former to one of the latter), it is soluble. The solution contains  $\text{SbCl}_3$ .

4.  $\text{H}_2\text{O}$ ; white "powder of algaroth" ( $\text{SbOCl}$ ). The precipitate dissolves in  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ . Accordingly, if this acid be present, water fails to produce a precipitate.

5.  $\text{H}_2\text{S}$ ; orange-red coloured precipitate ( $\text{Sb}_2\text{S}_3$ ).

6.  $(\text{NH}_4)_2\text{S}$ ; orange-red coloured precipitate, soluble in excess of the precipitant, but is again precipitated by  $\text{HCl}$ .

7.  $\text{NH}_3$ ; white  $\text{Sb}(\text{OH})_3$ , insoluble in excess.

8.  $\text{Zn}$ ; metallic Sb. If the reduction be performed in

a piece of platinum foil, or on a capsule, the Sb deposits on the platinum in black spots, difficult to remove.

## XXIX. ARSENIC, As, and ARSENIC ANHYDRIDE, $\text{As}_2\text{O}_3$ .

1. Arsenic heated before the blowpipe, gives off white fumes of  $\text{As}_2\text{O}_3$ . A smell resembling garlic may be perceived.

2. When heated in a tube, arsenic sublimes, giving a brilliant metallic mirror.

3. Metallic arsenic, heated with a small quantity of  $\text{HNO}_3$ , gives  $\text{As}_2\text{O}_3$ , which is but slightly soluble. If heated with an excess,  $\text{H}_3\text{AsO}_4$  is formed, easily soluble in water.

4.  $\text{As}_2\text{O}_3$ ; when heated in a glass tube sublimes, and deposits, in the cool part of the tube, as crystals, which may be easily seen by aid of a lens.

5. If  $\text{As}_2\text{O}_3$  be heated in a tube, with a fragment of charcoal, it is reduced, and a mirror of As is deposited in the cool part of the tube.

6.  $\text{As}_2\text{O}_3$  heated on charcoal sublimes; and gives off a smell resembling garlic; when volatilized from a porcelain crucible, no smell is perceptible.

$\text{As}_2\text{O}_3$  is soluble in water, but with difficulty.

7.  $\text{H}_2\text{S}$  added to this solution, gives a yellow colouration. On addition of HCl, yellow  $\text{As}_2\text{S}_3$  is precipitated, which is soluble in  $\text{NH}_3$  and  $(\text{NH}_4)_2\text{S}$ , but may be again precipitated by adding an acid.

8.  $(\text{NH}_4)_2\text{S}$  gives no precipitate; but on adding HCl, a yellow precipitate ( $\text{As}_2\text{S}_3$ ) is produced.

9.  $\text{AgNO}_3$  gives no precipitate; but on addition of a drop of  $\text{NH}_3$ ,  $\text{Ag}_3\text{AsO}_3$  is thrown down, which dissolves in excess of  $\text{NH}_3$ , and in  $\text{HNO}_3$ .

$\text{As}_2\text{O}_3$  dissolves, when boiled with  $\text{HNO}_3$ .

10.  $\text{H}_2\text{S}$  gives, in a lukewarm solution, when an excess is employed, yellow  $\text{As}_2\text{S}_3$ , and free S.

11.  $\text{AgNO}_3$  gives no precipitate. If the liquid be

carefully neutralised with  $\text{NH}_3$ , a reddish-brown precipitate of  $\text{Ag}_3\text{AsO}_4$  is produced, which is easily soluble in  $\text{NH}_3$ , and in  $\text{HNO}_3$ .

12. On addition of excess of  $\text{NH}_3$ ,  $\text{NH}_4\text{Cl}$ , and  $\text{MgSO}_4$ , a white precipitate ( $\text{Mg}[\text{NH}_4]\text{AsO}_4$ , analogous to that produced by phosphoric acid (see VII. 5), is formed.

### XXX. POTASSIC CHLORATE, $\text{KClO}_3$ .

1. When heated in a tube,  $\text{KClO}_3$  fuses, and gives off oxygen (*vide* XVII. 1). The residue contains  $\text{KCl}$ . It is dissolved in water and  $\text{AgNO}_3$  added, when a curdy precipitate ( $\text{AgCl}$ ) is formed (*vide* XXVI. 7).

2. If heated on charcoal, deflagration ensues.

3. If a small quantity of  $\text{KClO}_3$  be treated with concentrated  $\text{H}_2\text{SO}_4$ , and the mixture gently heated, a yellow explosive gas ( $\text{ClO}_2$ ) is evolved; the  $\text{KClO}_3$ , at the same time, assuming a yellow colour ( $3\text{KClO}_3 + 2\text{H}_2\text{SO}_4 = 2\text{ClO}_2 + \text{KClO}_4 + 2\text{KHSO}_4 + \text{H}_2\text{O}$ ).

4. If the dry salt be heated with  $\text{HCl}$ , a yellowish-green gas escapes—a mixture of  $\text{Cl}$  and  $\text{HClO}$ .

5.  $\text{KClO}_3$  is more easily soluble in hot than in cold water. In this solution neither  $\text{AgNO}_3$  nor  $\text{Hg}_2(\text{NO}_3)_2$  produce a precipitate.

6. Add a few drops of indigo-solution, some  $\text{H}_2\text{SO}_4$ , and then an aqueous solution of  $\text{SO}_2$  drop by drop. The colour of the indigo disappears, owing to the formation of lower oxides of chlorine.

### XXXI. SODIC SILICATE.

(Soluble Glass.)

1. The aqueous solution of soluble glass, if treated with an acid ( $\text{HCl}$ ,  $\text{HNO}_3$ ), or with  $\text{NH}_4\text{Cl}$ , or  $(\text{NH}_4)_2\text{CO}_3$ , deposits silicic acid ( $\text{Si}[\text{OH}]_4$ ).

2. This silicic acid may be rendered insoluble, if the



soluble glass be acidified with HCl, and evaporated to dryness. A few drops of HCl are added to the residue, then some water, and the fluid is boiled.

3. The silicic anhydride (silica,  $\text{SiO}_2$ ), which separates out, is collected on a filter. If heated with microcosmic salt, before the blowpipe, an opaque bead is produced.

4. Evaporate the solution left after the removal of the  $\text{SiO}_2$ . Nothing remains but NaCl (*vide* I).

### XXXII. HYDRIC POTASSIC OXALATE, $\text{KHC}_2\text{O}_4 + \text{H}_2\text{O}$ .

1. When heated in a tube, water is evolved; the mass blackens, and when the temperature has risen sufficiently, a gas is evolved (CO), which burns with a blue flame. The residue consists of  $\text{K}_2\text{CO}_3$ , exhibits an alkaline reaction, and effervesces with acids.

2. Some  $\text{KHC}_2\text{O}_4$  is mixed with  $\text{MnO}_2$  in a tube, and  $\text{H}_2\text{SO}_4$  added.  $\text{CO}_2$  is evolved with effervescence.

3.  $\text{CaCl}_2$ , added to the aqueous solution, produces a white precipitate ( $\text{CaC}_2\text{O}_4$ ). *Vide* X. 8.

4.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ; white precipitate of  $\text{PbC}_2\text{O}_4$ , soluble in  $\text{HNO}_3$ .

5. If the dry salt be heated in a tube with concentrated  $\text{H}_2\text{SO}_4$ , a mixture of gases escapes, which renders lime-water turbid ( $\text{CO}_2$ ), and burns with a blue flame (CO).

### XXXIII. SODIC HYPOSULPHITE, $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$ .

1. When heated in a tube, water is evolved. The residue consists of  $\text{Na}_2\text{S}$  and  $\text{Na}_2\text{SO}_4$ ; it is dissolved in water, and to a part of the solution some HCl is added.  $\text{H}_2\text{S}$  escapes; and if some  $\text{BaCl}_2$  be added,  $\text{BaSO}_4$  is precipitated. To another portion of the solution some  $\text{AgNO}_3$  is added; a black precipitate is thrown down ( $\text{Ag}_2\text{S}$ ). To the remainder of the solution, add some sodic nitro-prusside  $\text{Na}_2\text{Fe}(\text{NO})(\text{CN})_5$ ; violet colouration.

2. On addition of HCl to the aqueous solution, sulphur

is precipitated; the precipitation is accelerated by heat.  $\text{SO}_2$  may be recognised at the same time by its pungent smell.

3.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ; white precipitate ( $\text{PbS}_2\text{O}_3$ ), which blackens in boiling, owing to the formation of  $\text{PbSPbS}_2\text{O}_3 + \text{H}_2\text{O} = \text{PbS} + \text{H}_2\text{SO}_4$ .

4.  $\text{BaCl}_2$ ; white precipitate ( $\text{BaS}_2\text{O}_3$ ), soluble in a large amount of water.

5.  $\text{FeCl}_3$ ; violet colouration, disappearing in a short time.

### XXXIV POTASSIC IODIDE, KI.

1.  $\text{AgNO}_3$  added to the aqueous solution, gives a yellow precipitate of KI, which is soluble in  $\text{NH}_3$  and  $\text{HNO}_3$ .

2.  $\text{HgCl}_2$ ; red precipitate of  $\text{HgI}_2$ .

3.  $\text{Hg}_2(\text{NO}_3)_2$ ; greenish-yellow,  $\text{Hg}_2\text{I}_2$ .

4.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ ; yellow  $\text{PbI}_2$ , soluble in a considerable quantity of boiling water. On cooling, glittering crystals are deposited.

5. The solution is acidified with a few drops of  $\text{H}_2\text{SO}_4$ , some starch solution added, and then  $\text{KNO}_2$  (potassic nitrite), drop by drop. The liquid assumes a deep blue colour. ( $\text{KI} + \text{H}_2\text{SO}_4 + \text{KNO}_2 = \text{I} + \text{NO} + \text{K}_2\text{SO}_4 + \text{H}_2\text{O}$ ).

6. Some  $\text{K}_2\text{Cr}_2\text{O}_7$  and concentrated  $\text{H}_2\text{SO}_4$  are added to the solution, and iodine is set free, which escapes as violet vapours when the solution is boiled.

7. Boil the solution with  $\text{FeCl}_3$ , iodine is given off. ( $\text{FeCl}_3 + \text{KI} = \text{I} + \text{FeCl}_2 + \text{KCl}$ ).

### XXXV. STRONTIC SULPHATE, $\text{SrSO}_4$ .

(*Celestin.*)

1.  $\text{SrSO}_4$  is not altered by heat.

2. If heated on charcoal, before the blowpipe, with  $\text{Na}_2\text{CO}_3$ , strontic sulphide is formed, which is insoluble in water and in acids.

3. If  $\text{SrSO}_4$  be boiled with a solution of  $\text{Na}_2\text{CO}_3$ , and

filtered while boiling, some  $\text{HCl}$  added to the filtrate, and then  $\text{BaCl}_2$ , a precipitate of  $\text{BaSO}_4$  is thrown down.

4. The precipitate, insoluble in  $\text{Na}_2\text{CO}_3$ , which consists of  $\text{SrCO}_3$ , is dissolved by pouring some  $\text{HCl}$  through the filter.  $\text{CaSO}_4$  added to the filtrate, produces a turbidity after lapse of time.

5. If  $\text{SrSO}_4$  be boiled with a solution of two parts of  $\text{K}_2\text{CO}_3$  and one part of  $\text{K}_2\text{SO}_4$ , and the liquid filtered while boiling; the residue after being washed out with  $\text{H}_2\text{O}$ , dissolved in  $\text{HCl}$ , and evaporated to dryness; some alcohol added to the residue ( $\text{SrCl}_2$ ), and ignited; a crimson-red colour is imparted to the flame.

### XXXVI. BARIUM SULPHATE, $\text{BaSO}_4$ .

(*Heavy spar.*)

1. When heated in a tube  $\text{BaSO}_4$  remains unaltered.

2. Heated with  $\text{Na}_2\text{CO}_3$ , on charcoal, before the blow-pipe, a sulphide is produced.  $\text{BaSO}_4$  is insoluble in  $\text{H}_2\text{O}$ , and in acids.

3. If  $\text{BaSO}_4$  be repeatedly boiled with successive quantities of  $\text{Na}_2\text{CO}_3$ , and the solution filtered while boiling, it may be decomposed. The solution contains  $\text{Na}_2\text{SO}_4$  (*vide* XXXV. 3).

4. The insoluble ( $\text{BaCO}_3$ ) may be dissolved by pouring  $\text{HCl}$  through the filter in which it has been collected.  $\text{CaSO}_4$  added to the solution produces an instantaneous precipitate ( $\text{BaSO}_4$ ).

5. The mixture of  $\text{K}_2\text{CO}_3$  and  $\text{K}_2\text{SO}_4$  does not act decomposingly on  $\text{BaSO}_4$  (*vide* XXXV. 5). The residue, filtered off, and treated with  $\text{HCl}$ , does not dissolve.

### XXXVII. POTASSIC CYANIDE, KCN.

1.  $\text{HCl}$  added to the aqueous solution liberates hydrocyanic acid (prussic acid,  $\text{HCN}$ ), which may be recognised

by its smell, resembling bitter almonds. It is excessively poisonous.

2.  $\text{AgNO}_3$ ; white, curdy precipitate ( $\text{AgCN}$ ), easily soluble in excess of  $\text{KCN}$ , and in  $\text{NH}_3$ , but insoluble in  $\text{HNO}_3$ .

3. Add to the solution of  $\text{KCN}$ ,  $\text{FeCl}_3$ ,  $\text{FeSO}_4$ , and  $\text{NaOH}$ , till a precipitate is formed, and heat gently. After the liquid is acidified with  $\text{HCl}$ , the ferric oxide dissolves, and a precipitate of "Prussian blue" remains undissolved ( $3\text{Fe}[\text{CN}]_2 \cdot 4\text{Fe}[\text{CN}]_3$ ).

4. This precipitate, after being filtered and washed with water, is removed to a test-tube, and treated with  $\text{NaOH}$ .  $\text{Fe}(\text{OH})_3$  is precipitated, and the solution contains  $4\text{KCN} \cdot \text{Fe}(\text{CN})_2$ ; after again acidifying with  $\text{HCl}$ , however, Prussian blue is again thrown down.

### XXXVIII. PLUMBIC ACETATE, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 + 3\text{H}_2\text{O}$ .

1. When heated in a tube, water is given off; then  $\text{HC}_2\text{H}_3\text{O}_2$  volatilises. When a higher heat is applied, only carbon and metallic lead remain behind.

2. If heated on charcoal, before the blowpipe, with  $\text{Na}_2\text{CO}_3$ , a malleable metallic bead is formed, and the charcoal is coated with a yellow incrustation ( $\text{PbO}$ ).

3. Heat in a tube, with concentrated  $\text{H}_2\text{SO}_4$ ;  $\text{HC}_2\text{H}_3\text{O}_2$  is liberated, which may be recognised by its smell, and by its action on litmus-paper.

4. If some alcohol be added to crystals of the salt in a test-tube, an equal volume of concentrated  $\text{H}_2\text{SO}_4$  added, and the mixture heated, acetic ether ( $\text{C}_2\text{H}_5\text{O} \cdot \text{C}_2\text{H}_3\text{O}$ ) is evolved, which may be identified by its pleasant fruity odour.

# SYSTEMATIC COURSE OF ANALYSIS.

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## A.—PRELIMINARY EXAMINATION.

### I. Heat some of the Substance in a Test-tube.

If a liquid, a portion must be evaporated to dryness, and submitted to the following tests:—

1. *Water is evolved*, which has either an alkaline ( $\text{NH}_3$ ), an acid ( $\text{HCl}$ ,  $\text{SO}_2$ , etc.), or a neutral reaction (water of crystallisation).

2. *A sublimate is formed*: reddish-yellow drops solidifying to a yellow-coloured mass on cooling (S). A *white* sublimate may consist of ammonium salts,  $\text{HgCl}_2$ ,  $\text{Hg}_2\text{Cl}_2$ ,  $\text{As}_2\text{O}_3$ ,  $\text{Sb}_2\text{O}_3$ . A *yellow* sublimate may be  $\text{HgI}_2$ , or  $\text{As}_2\text{S}_3$ . *Black* sublimate;  $\text{HgS}$ , which, when rubbed, assumes a red tint. *Metallic mirror*, As; *small metallic globules*, Hg.

3. *Evolution of gas*. Oxygen; substances containing  $\text{HClO}_3$ , and  $\text{HNO}_3$ .—Some metallic oxides—*Carbonic oxide*; oxalates.—*Carbonic anhydride*; carbonates (the gas renders baryta-water turbid).—*Ammonia*; ammonium salts.—*Sulphurous anhydride*; easily recognisable by its smell, and acid reaction.—*Hydric sulphide*.—*Cyanogen*; has a penetrating odour, and burns with a red flame.—*Nitric peroxide* ( $\text{NO}_2$ ) nitrates; reddish-coloured gas.

4. *The substance fuses*; presence of alkaline salts.

5. *The substance does not fuse, but changes colour*.—The substance, originally white, turns yellow, but on cooling

reassumes its white colour; ZnO. The substance is white; on being heated it turns dark yellow, and on again cooling assumes a pale yellow colour; SnO<sub>2</sub>. White or light yellow, when heated dark yellow; on cooling, light yellow; Bi<sub>2</sub>O<sub>3</sub> (fusible). White or yellow, changing to dark red when heated, and when cold again yellow; PbO (fusible).

6. *The substance carbonises*; presence of organic compounds.

7. *The substance remains unaltered*; BaCO<sub>3</sub>, BaSO<sub>4</sub>, etc.

## II. Heat the Substance before the Blowpipe on Charcoal.

1. *Deflagration*; chlorates, nitrates.

2. *The substance fuses, penetrates into the pores of the charcoal, or forms a transparent glass*; salts of K, Na, Li, or (NH<sub>4</sub>). The residue has an alkaline reaction; carbonates, nitrates.

3. *The substance is infusible*; compounds of the alkaline earths, SiO<sub>2</sub>, ZnO, etc.

4. *The substance becomes incandescent*; ZnO, MgO, CaO, SrO. If a smell be observed, during the last two experiments, As<sub>2</sub>O<sub>3</sub> (recognisable by its odour of garlic), SO<sub>2</sub> or NH<sub>3</sub> may be present.

5. Moisten the infusible residue, when cold, with solution of *cobaltic nitrate*, and ignite again. Al<sub>2</sub>O<sub>3</sub>; dark blue colour. MgO; pale flesh colour. BaO, SrO, and CaO; grey colour. ZnO; yellowish-green. SnO<sub>2</sub>; bluish-green. Sb<sub>2</sub>O<sub>3</sub>; dirty, dark-green colour.

## III. Heat the Substance mixed with Na<sub>2</sub> CO<sub>3</sub> before the Blowpipe on Charcoal.

1. If a *sulphide* is formed (recognisable by its action on a silver coin, see p. 10), the presence of S may be inferred

2. *Gray metallic powder, without incrustation*; Fe, Ni or Co (magnetic).

3. *Metallic bead, without incrustation*; red, Cu; the flame has at the same time a greenish colour. White, Ag; sometimes accompanied by a slight red incrustation, Sn; it is more easily reduced if borax, or, better, if KCN be mixed with the  $\text{Na}_2\text{CO}_3$ . If heated for a length of time, the metallic beads are changed into yellow  $\text{SnO}_2$ , which is not volatile, and on cooling changes to a white colour. Yellow; Au.

4. *Metallic bead, with incrustation*; I. The metallic bead is ductile, the incrustation yellow; Pb. When the deposit is heated in the reducing flame, it volatilises, colouring the flame blue. II. The metallic bead is brittle. A *white* volatile incrustation shows the probable presence of Sb (*vide* XXVIII. 2). A yellow incrustation, darkening in colour when heated, is produced by Bi. It is lighter in colour than that of Pb, and volatile when exposed to the reducing flame, to which it does not impart a colour.

5. *Incrustation without metallic bead*; white, very volatile, with a smell of garlic. As; white, yellow while hot. Zn; reddish-brown. Cd; most easily observed after cooling.

#### IV. Colouration of the Flame.

Heat a portion of the substance, moistened with HCl, on a piece of platinum wire, in the outer flame of a bunsen burner, or in the oxidising flame of the blowpipe.

*Yellow*; Na (I. 2).

*Red*; Sr, Li, Ca (yellowish-red).

*Green*; Ba (yellowish-green), (Tl), Cu.

*Blue*; Cu  $\text{Cl}_2$ .

*Violet*; K (IV. 3).

V. Colour of a Bead of Borax, or Microcosmic Salt  
 ( $\text{PO}_4\text{HNaNH}_4 + 4\text{H}_2\text{O}$ ; when fused,  $\text{PO}_2\text{ONa}$ ).

	OXIDISING FLAME.		REDUCING FLAME.			
		Hot.	Cold.		Hot.	Cold.
Green.	Bright green	Cr Ur Cu	Cr Ur	In sufficient quantity	Cr Ur Fe	Cr Ur
Blue.		Co	Co Cu		Co	Co
Yellow.	When much is present, opal coloured	Ag Bi Ur Pb Cd Ti	Ag		Ti	
	If present in sufficient quantity		Fe Ni		Ni	Ni
Red.	Brownish-red	Fe Ni		Brownish-red, opaque		Cu
Violet.				After addition of tin-foil		Ti
Gray (reduced metal).				After addition of tin-foil	Ag Pb Ni	Ag Pb Bi Ni
Insoluble.		$\text{SiO}_2$	$\text{SiO}_2$		$\text{SiO}_2$	$\text{SiO}_2$



## VI. Solution of the Substance.

1. *The substance is solid, but not a metal.*—Try if it is soluble in *water*. If the substance is completely, or in great part soluble, treat a larger quantity, 1 to 3 grammes, in a similar manner, and investigate the aqueous solution. If, on the contrary, the substance is insoluble, or soluble with difficulty, evaporate a portion of the filtered fluid on platinum-foil, and notice if a residue is left on evaporation, and if it is stable, volatile, or non-volatile. If the residue is extremely small, the substance may be considered to be insoluble in water, and should be tested in a similar manner as to its solubility in dilute  $\text{HCl}$ . Notice if during the action of  $\text{HCl}$ ,  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ , or  $\text{HCN}$ , is evolved.

If the substance does not dissolve in dilute  $\text{HCl}$ , even on boiling, it must be treated with **concentrated hydrochloric acid**. (Evolution of  $\text{Cl}$  betokens the presence of higher oxides or chromates). The insoluble in  $\text{HCl}$  must be treated with  $\text{HNO}_3$  in the same manner. Should there still remain an insoluble residue, an attempt must be made to dissolve it in a mixture of three volumes of  $\text{HCl}$ , and one of  $\text{HNO}_3$  (“*aqua regia*.”)

*For substances insoluble in acids, vide sec. 51, p. 54.*

2. *The substance is a fluid.*—Test the fluid with litmus-paper. Evaporate a portion to dryness, and proceed to test it as described in I., II., III., IV., and V.

3. *The substance is a metal.*—It is treated with  $\text{HNO}_3$ ;  $\text{Au}$  and  $\text{Pt}$  remain unaltered, and may be dissolved in *aqua regia*.  $\text{Sb}$  and  $\text{Sn}$  (sometimes containing  $\text{As}$ ) remain as white oxides, insoluble both in water and in  $\text{HNO}_3$ . It is filtered off, and, after being thoroughly washed, is treated with  $\text{HCl}$  (sec. 49), or with  $\text{Na}_2\text{S}$  (sec. 50).

4. Endeavour to obtain as strong solutions as possible. A large excess of acid, especially of  $\text{HNO}_3$ , is to be avoided, but may be usually removed by evaporation. Sometimes, however, evaporation may lead to loss, as in the instance of arsenic, existing as  $\text{AsCl}_3$ , which volatilises on evaporation.

5. It is advisable to investigate the aqueous and the acid solutions separately; as in the former, alkalis, nitrates, chlorates, and many sulphates are present, but no baric, strontic, or calcic phosphates, which latter are soluble in acids. Alkalis are seldom met with in the acid solution. The solutions in HCl and HNO<sub>3</sub>, may be mixed, and the precipitate, if one is formed, collected, and investigated by itself.

### B.—DETECTION OF BASES.

The solution of the substance, if aqueous, is acidified with HCl; if acid, it is diluted with H<sub>2</sub>O; should a precipitate occur, no notice is taken of it. H<sub>2</sub>S is added to the fluid; the resulting precipitate contains all metals of groups V. and VI.

To the filtrate is added an excess of NH<sub>3</sub>, and a quantity of (NH<sub>4</sub>)<sub>2</sub>S, sufficient to precipitate groups III. and IV.

A portion of the filtrate is tested for Ba, Sr, Ca, and Mg, by means of NH<sub>3</sub>, and Na<sub>2</sub>HPO<sub>4</sub> (group II.)

#### General Behaviour of the Metals with Reagents.

H <sub>2</sub> S		(NH <sub>4</sub> ) <sub>2</sub> S		Na <sub>2</sub> HPO <sub>4</sub>	No Precipitate
Gives a precipitate in acid solution. The precipitate is treated with (NH <sub>4</sub> ) <sub>2</sub> S.		Precipitates in ammoniacal solution as		Precipitates	with the former reagents.
Soluble	Insoluble	Hydrate	Sulphide		
As	Hg	Al	Ni	Ba	K
Sb	Ag	Cr	Co	Sr	Na
Sn	Pb		Fe	Ca	(NH <sub>4</sub> )
	Bi		Mn	Mg	Li
	Cu	Phosphates and oxalates of Ba, Sr, Ca, and Mg (Ur)			
	Cd		Zn		
VI.	V.	IV.	III.	II.	I.

## GROUP I.—ALKALIES, K, Na, (NH<sub>4</sub>), (Li).

1. The fluid, in which H<sub>2</sub>S and (NH<sub>4</sub>)<sub>2</sub>S failed to produce a precipitate, or from which the metals belonging to groups III.-VI. are already removed, and provided Ba, Sr, Ca, and Mg are absent, is evaporated to dryness, and ignited to volatilise the salts of ammonium. Residue: K, Na, Li.

2. Heat a portion of the dry residue on the loop of a platinum wire in the outer flame of a bunsen burner, or on the blowpipe flame.

*Yellow colouration* of the flame; Na. Apply the tests given in I. 2 (p. 3).

*Violet*; K. In presence of Na, the violet colour is apparent only when viewed through an indigo prism (*vide* IV. 3, p. 5).

*Red*; Li. The colouration which Li gives to the flame is also observed in presence of much Na. It may be identified most easily by means of the spectroscope. The separation of Li from K and Na may be most easily accomplished, by the solubility of LiCl in a mixture of absolute alcohol and ether, in which KCl and NaCl do not dissolve. A concentrated aqueous solution of a lithium salt gives, when treated with (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, a white precipitate of Li<sub>2</sub>CO<sub>3</sub>.

3. *Detection of NH<sub>3</sub>*.—A portion of the original substance is treated with NaOH, and heated slightly. The NH<sub>3</sub> may be recognised by its smell, by its alkaline reaction on moistened test paper, and by its giving white fumes when a rod, moistened with acetic acid or HCl, is held near it.

## GROUP II. Ba, Sr, Ca, Mg.

4. If, in a small portion of the fluid in which (NH<sub>4</sub>)<sub>2</sub>S has failed to give a precipitate, or which has been filtered off from it, and which has been treated as directed in

sec. 10, p. 41, a precipitate is formed on addition of  $\text{NH}_3$  and  $\text{Na}_2\text{HPO}_4$ , another portion of the same fluid is treated with solution of *calcic sulphate*.

Immediate turbidity : Ba (compare sec. 10).

5. Add a solution of one part of  $(\text{NH}_4)_2\text{SO}_4$  dissolved in four parts water to the remainder of the fluid, in which the metals of groups I. and II. may be present. If a precipitate is formed, add excess of  $(\text{NH}_4)_2\text{SO}_4$ , and boil for a short time. The precipitate consists of  $\text{BaSO}_4$  and  $\text{SrSO}_4$  (sec. 6); in the filtrate Ca, Mg, and the alkalies (sec. 7) are present (comp. sec. 10).

6. The *precipitate* of  $\text{BaSO}_4$  and  $\text{SrSO}_4$  is filtered off, and, after being thoroughly washed, boiled with a mixture of  $\text{K}_2\text{CO}_3$ , and  $\text{K}_2\text{SO}_4$  (*vide* XXXV. 5, and XXXVI. 5, p. 31). It is then filtered, the precipitate *carefully* washed, and treated on the filter with HCl.  $\text{SrCO}_3$  dissolves with effervescence, and its presence may be confirmed with solution of  $\text{CaSO}_4$ , or by the flame colouration (*vide* XXXV. 4, 5, p. 31).

7. The filtrate from  $\text{BaSO}_4$ , and  $\text{SrSO}_4$ , or the fluid in which  $(\text{NH}_4)_2\text{SO}_4$  produced no precipitate, is treated with  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ . Precipitate : Ca. A part of the filtrate from the  $\text{CaC}_2\text{O}_4$ , or of the fluid in which  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  failed to produce a precipitate, is treated with  $\text{NH}_3$ , and  $\text{Na}_2\text{HPO}_4$ . Precipitate : Mg. If Mg is not present, proceed with the remainder of the fluid, as directed in sec. 1.

8. **Magnesia is Present.**—The solution from which Ba, Sr, and Ca have been removed, and of which a small portion has been taken in order to test for Mg, is evaporated to dryness, and ignited to drive off the salts of ammonium. The residue is dissolved in water; lime-water or baryta-water, which contains no alkaline impurity, is added till the liquid has an alkaline reaction; it is then boiled and filtered off from the mixture of MgO, and excess of CaO. The precipitate is washed, the filtrate added to the washings, and concentrated by evaporation; the CaO, which still remains in solution, is removed by

$\text{NH}_3$  and  $(\text{NH}_4)_2\text{CO}_3$  (or  $[\text{NH}_4]_2\text{C}_2\text{O}_4$ ), or the  $\text{BaO}$  precipitated with  $\text{H}_2\text{SO}_4$ . The filtrate from this precipitate is evaporated to dryness.

Detection of  $\text{NH}_3$ : *vide* sec. 3. Residue : Na, K (*vide* sec. 2).

### GROUPS III. and IV.; Al, Cr, Ni, Co, Fe, Mn, Zn, (Ur).

9. To a portion of the solution, in which  $\text{H}_2\text{S}$  failed to produce a precipitate, or from which the V. and VI. groups precipitated by that reagent have been removed by filtration,  $\text{NH}_3$  is added till an alkaline reaction is produced ; then  $(\text{NH}_4)_2\text{S}$ . If a precipitate is formed, the remainder of the solution is treated in the same way. Warm gently, filter off the precipitate, and wash it with water containing  $(\text{NH}_4)_2\text{S}$ , in order to prevent the sulphides oxidising. If Ni be present, the filtrate is usually dark brown and turbid from dissolved NiS.

10. Unite the filtrate with the washings, test for the presence of Ba, Sr, Ca, and Mg, and treat it further, as directed in sec. 5 or sec. 1, as the case may be. Since an excess of  $(\text{NH}_4)_2\text{S}$  may be prejudicial to the reactions for detecting Ba or Sr, the filtrate from the precipitate obtained by  $(\text{NH}_4)_2\text{S}$  ought to be evaporated to half its original bulk, acidified with a little  $\text{HCl}$ , and boiled in order to destroy any  $(\text{NH}_4)_2\text{S}_2\text{O}_3$  which may possibly have been formed, and the solution filtered. To a portion of the filtrate a solution of  $\text{CaSO}_4$  is added for the detection of Ba and Sr. (Turbidity after some time, sec. 4). The rest of the filtrate is rendered alkaline with  $\text{NH}_3$ ,  $(\text{NH}_4)_2\text{SO}_4$  added, and treated as described in sec. 5 for the separation of Ba and Sr.

11. The precipitate of groups III. and IV. is well washed, removed from the filter by help of a spatula, and placed in a flask. Dilute  $\text{HCl}$  is then added, and a gentle heat applied. If a black insoluble residue remains, Co and Ni (sec. 12) are present. If the insoluble

be *white*, and in small quantity, it is *sulphur*. It is filtered off, and investigated as described in sec. 13.

12. *Separation of Co and Ni.*—Part of the precipitate, which must have been well washed, is heated in a bead of borax or microcosmic salt before the blowpipe (XX. 3, p. 21). The rest of the black sulphides is dissolved in concentrated  $\text{HNO}_3$ ; the solution concentrated by evaporation, and neutralised with  $\text{Na}_2\text{CO}_3$ . An excess of  $\text{KNO}_2$  is added, the fluid acidified with a few drops of  $\text{HC}_2\text{H}_3\text{O}_2$ , and allowed to stand for twenty-four hours. The yellow precipitate of Co (XX. 4, p. 21) is filtered off; the filtrate treated again with  $\text{KNO}_2$  to ensure the removal of all Co, and the Ni finally precipitated with  $\text{NaOH}$  as  $\text{Ni}(\text{OH})_2$  (p. 20).

Nickel may be detected in presence of cobalt by dissolving the mixed sulphides in concentrated  $\text{HNO}_3$ , and neutralising with  $\text{Na}_2\text{CO}_3$ ;  $\text{KCN}$  is then added till the resulting precipitate has dissolved, and  $\text{NaClO}$  till the fluid smells strongly of it, even after being shaken. It is then boiled. Black precipitate;  $\text{Ni}_2\text{O}_3$ .

13. The solution of the remainder of the metals of groups III. and IV. in  $\text{HCl}$ , is boiled till all traces of  $\text{H}_2\text{S}$  are removed; and if iron be present (its presence is betrayed by the black colour of the precipitate by  $[\text{NH}_4]_2\text{S}$ ), it must be oxidised by being boiled with  $\text{HNO}_3$ , or  $\text{HClO}_3$ . The further process depends on the presence or absence of Cr. If Cr be present, the hydrochloric acid solution (sec. 11) has a violet or green colour. Chromium will also have been detected in the preliminary examination, from the colour of the borax bead.

### Chromium is Absent.

14. *The precipitate with  $(\text{NH}_4)_2\text{S}$  had a light colour:* Al, Mn, Zn; Fe is absent. To the solution in  $\text{HCl}$  (sec. 13)  $\text{NaOH}$  is added in excess. Precipitate Mn. Examine the precipitate before the blowpipe, as directed in XVII. 9, p. 19. The filtrate from  $\text{Mn}(\text{OH})_2$  is divided into two

portions. The one is acidified with HCl, and excess of  $\text{NH}_3$  added. Precipitate: Al.  $\text{H}_2\text{S}$  is added to the other portion. Precipitate Zn.

15. *The precipitate with  $(\text{NH}_4)_2\text{S}$  was dark-coloured.* Iron is present.

Proceed as directed in sec. 25, or add to the solution in HCl (sec. 13)  $\text{Na}_2\text{CO}_3$  till a permanent precipitate is produced; dissolve it in a small quantity of  $\text{HC}_2\text{H}_3\text{O}_2$ . Some  $\text{NaC}_2\text{H}_3\text{O}_2$  is then added, and it is boiled till the supernatant fluid is colourless. If it has a brown, or yellow colour, either  $\text{NaC}_2\text{H}_3\text{O}_2$  has not been added in sufficient quantity, or it has not been long enough boiled.

16. *Precipitate*; Al, Fe (sec. 16); *Filtrate* Mn, Zn (sec. 17). The precipitate of Al and Fe, after being washed, is dissolved in HCl; an excess of NaOH is then added, and the fluid boiled.

*Precipitate*; Fe. Confirm by the tests given in XVI. 9, 10, p. 18. The filtrate from the precipitate of  $\text{Fe}(\text{OH})_3$  is acidified with HCl, and the  $\text{Al}(\text{OH})_3$  precipitated by adding excess of  $\text{NH}_3$ .

17. The filtrate, which contains Mn and Zn, is treated with  $\text{H}_2\text{S}$ . Precipitate, Zn.

The filtrate from  $\text{ZnS}$ , which must contain an excess of  $\text{H}_2\text{S}$ , is rendered alkaline by  $\text{NH}_3$ , and some  $(\text{NH}_4)_2\text{S}$  added. *Flesh-coloured precipitate*; Mn.

### Chromium is Present.

The original solution has a green or violet colour, provided a chromic salt is present, and yellow or red if a chromate be present.

18. The solution in HCl (sec. 13), which has been heated with  $\text{KClO}_3$ , or with  $\text{HNO}_3$ , must be carefully neutralised with  $\text{Na}_2\text{CO}_3$ , care being taken that no permanent precipitate be formed; when the fluid is *thoroughly cold*, add some  $\text{BaCO}_3$ , kept in suspension by water, *without heating*. Shake the fluid, let it stand for some time in the cold, and filter as soon as the supernatant fluid be-

comes colourless. The precipitate may contain Al, Fe, Cr (sec. 19); the filtrate Mn, Zn (sec. 23).

19. The precipitate, after being well washed, is dissolved in HCl, and the excess of  $\text{BaCO}_3$  neutralised with  $\text{H}_2\text{SO}_4$ . The liquid is boiled, and filtered;  $\text{NH}_3$  is added to the filtrate: *Precipitate*:  $\text{Fe}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ . This precipitate is filtered off, washed, removed from the filter, and dissolved in a small quantity of  $\text{HNO}_3$  in a basin. Some solid  $\text{KClO}_3$  is added to the solution, which is boiled for a few minutes, more being added, if necessary, till it assumes a yellowish-red colour. Excess of NaOH is now added, and the fluid is heated. *Precipitate*: Fe (sec. 20). *Filtrate*: Cr, Al (sec. 21, 22).

20. **Fe.** The precipitate of  $\text{Fe}_2\text{O}_3^*$  is filtered off and tested, as in sec. 16.

21. **Al.** The alkaline filtrate from  $\text{Fe}_2\text{O}_3$  is acidified with  $\text{HNO}_3$ , and precipitated with  $\text{NH}_3$ ;  $\text{Al}_2\text{O}_3$ . To confirm aluminum, ignite the precipitate of  $\text{Al}_2\text{O}_3$  moistened with a few drops of  $\text{Co}(\text{NO}_3)_2$  on charcoal, before the blowpipe, as directed in II. 5, p. 34.

22. **Cr.** The filtrate, on removal of the precipitate of  $\text{Al}_2\text{O}_3$ , is yellow, owing to the presence of  $\text{CrO}_3$ . It is acidified with  $\text{HC}_2\text{H}_3\text{O}_2$ , and precipitated with  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ , as yellow  $\text{PbCrO}_4$ .

23. **Mn and Zn.** The filtrate from the precipitate formed by  $\text{BaCO}_3$  (sec. 18) is freed from the dissolved baryta by adding  $\text{H}_2\text{SO}_4$ , and Mn and Zn precipitated by adding NaOH to the boiling solution. A portion of the precipitate is tested for Mn by means of  $\text{KNO}_3$  and  $\text{Na}_2\text{CO}_3$

\* In all the above-given methods of separation uranium remains always with the iron, and is always precipitated as  $\text{Ur}_2\text{O}_3$  when the Fe is precipitated as  $\text{Fe}_2\text{O}_3$ . In order to separate **Ur** from **Fe**, the mixture of both is dissolved in HCl, exactly neutralised with  $\text{NH}_3$ , and the Fe precipitated by a mixture of  $(\text{NH}_4)_2\text{S}$ , and  $(\text{NH}_4)_2\text{CO}_3$ , as  $\text{FeS}$ . The filtrate is acidified with HCl, boiled, and oxidised with  $\text{HNO}_3$ . The  $\text{Ur}_2\text{O}_3$  is precipitated on addition of  $\text{NH}_3$ .

Confirm it by the blowpipe reactions, p. 36, or by solution in HCl, and addition of  $4\text{KCN}$ ,  $\text{FeCN}$ ; reddish-brown precipitate.



(XVII. 9, p. 19). The remainder of the precipitate is dissolved in  $\text{HC}_2\text{H}_3\text{O}_2$ , and  $\text{H}_2\text{S}$  passed through the solution. Precipitate:  $\text{ZnS}$ . The filtrate is tested for  $\text{Mn}$ , as in sec. 17.

24.  $\text{FeO}$ , and  $\text{Fe}_2\text{O}_3$ . As  $\text{Fe}$  is always precipitated as  $\text{Fe}_2\text{O}_3$ , in following the usual course of qualitative analysis, it is necessary, in testing for  $\text{FeO}$ , to use a portion of the *original* substance. Some  $3\text{KCN} \cdot \text{FeCN}_3$  is added to the acid solution of the original substance. If  $\text{FeO}$  be present, a blue precipitate will be thrown down— $3\text{FeCN}_2 \cdot 2\text{FeCN}_3$  (Turnbull's blue).  $\text{Fe}_2\text{O}_3$  is identified as in XVI. 9, 10.—These reactions are, of course, only practicable when substances, such as ferro, or ferridcyanides, or sulphocyanides, which would interfere with the reaction, are absent. In presence of  $\text{HNO}_3$ , or of  $\text{HCl}$  when the higher metallic oxides are present, or of  $\text{CrO}_3$ , etc.,  $\text{FeO}$  is oxidised into  $\text{Fe}_2\text{O}_3$ .

25. If it is wished to separate the metals of groups III. and IV., *in absence of Cr.* (sec. 18), proceed with the filtrate as directed in sec. 23 for the separation of  $\text{Mn}$  and  $\text{Zn}$ . The precipitate of  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , and excess of  $\text{BaCO}_3$  is dissolved in  $\text{HCl}$ , as in sec. 19, the excess of  $\text{BaCl}_2$ , removed by adding  $\text{H}_2\text{SO}_4$ , and excess of  $\text{NaOH}$  added to the filtrate. The presence of  $\text{Fe}$  and  $\text{Al}$  are confirmed as given in sec. 20, 21.

### Baric, Strontic, Calcic, or Magnesian Oxalates or Phosphates are present.

26.  $\text{H}_2\text{C}_2\text{O}_4$ . Part of the precipitate produced by  $(\text{NH}_4)_2\text{S}$  is boiled for some time with an excess of  $\text{Na}_2\text{CO}_3$ , and is filtered while boiling, acidified with  $\text{HC}_2\text{H}_3\text{O}_2$ , and  $\text{CaCl}_2$  added. Precipitate:  $\text{CaC}_2\text{O}_4$ .

27. *If  $\text{H}_2\text{C}_2\text{O}_4$  be present*, dry the remainder of the precipitate produced by  $(\text{NH}_4)_2\text{S}$ , and ignite it in a covered porcelain crucible till the oxalic acid is destroyed. The residue is dissolved in  $\text{HCl}$ , an excess of  $\text{NH}_3$  added and reprecipitated with  $(\text{NH}_4)_2\text{S}$ . The bases which were

formerly united to  $\text{H}_2\text{C}_2\text{O}_4$  remain in solution; they can be tested for in the filtrate, as in sec. 4. If  $\text{H}_2\text{C}_2\text{O}_4$  be absent, proceed with the rest of the  $(\text{NH}_4)_2\text{S}$  precipitate as directed in sec. 28.

28. The precipitate produced by  $(\text{NH}_4)_2\text{S}$ , now freed from oxalic acid, is digested with  $\text{HCl}$  (sec. 11). A black residue points to the presence of  $\text{Ni}$  and  $\text{Co}$  (sec. 12); the solution is treated with  $\text{HNO}_3$  or  $\text{HClO}_3$  in order to oxidise the  $\text{Fe}$  which may be present; in a small portion  $\text{Fe}$ , and  $\text{H}_3\text{PO}_4$  are tested for as in sec. 29, 30.

29.  $\text{Fe}$  may be tested for with  $\text{KCNS}$ , or  $4\text{KCN}.\text{FeCl}_2$ .

30.  $\text{H}_3\text{PO}_4$ . Some of the solution in  $\text{HCl}$  of the precipitate produced by  $(\text{NH}_4)_2\text{S}$ , is boiled to get rid of excess of  $\text{H}_2\text{S}$ ; it is then added, drop by drop, to a solution of ammonic molybdate, previously acidified with  $\text{HNO}_3$ . Yellow precipitate,  $\text{H}_3\text{PO}_4$  (sec. 59).

31. The remainder of the acid solution of the  $(\text{NH}_4)_2\text{S}$  precipitate is carefully neutralised with  $\text{Na}_2\text{CO}_3$  (if excess be added, the precipitate must be redissolved in a small quantity of  $\text{HC}_2\text{H}_3\text{O}_2$ ) and  $\text{NaC}_2\text{H}_3\text{O}_2$  added. If the fluid does not become red,  $\text{FeCl}_3$  must be added. It is boiled till the supernatant fluid is perfectly colourless; filtered while boiling, and well washed. The precipitate may consist of  $\text{Fe}$ ,  $\text{Al}$ ,  $\text{Cr}$ ,  $\text{H}_3\text{PO}_4$ , (sec. 32). The filtrate contains  $\text{Mn}$ ,  $\text{Zn}$ , and  $\text{Ba}$ ,  $\text{Sr}$ ,  $\text{Ca}$ , or  $\text{Mg}$ , formerly existing as phosphates\* (sec. 34).

32.  $\text{Al}_2\text{O}_3$ . The precipitate of  $\text{Al}$ ,  $\text{Cr}$ ,  $\text{Fe}$ ,  $\text{H}_3\text{PO}_4$ , after being filtered and washed, is boiled with excess of  $\text{NaOH}$ , and  $\text{Al}_2\text{O}_3$  tested for in the filtrate by addition of  $\text{HCl}$ , and  $\text{NH}_3$ , as in sec. 16.

\* If the substance contains  $\text{Al}_2\text{O}_3$ , and a phosphate,  $\text{AlPO}_4$  may have been formed, in precipitating with  $(\text{NH}_4)_2\text{S}$ , which would not be acted on by  $\text{FeCl}_3$ , as in sec. 31. On treating the precipitate with  $\text{NaOH}$  (sec. 32)  $\text{Al}(\text{OH})_3$  would not be dissolved, but  $\text{AlPO}_4$ , which would be again precipitated, on addition of  $\text{HCl}$  and  $\text{NH}_3$ . In order to test for  $\text{H}_3\text{PO}_4$ , the precipitate produced in the  $\text{NaOH}$  solution by  $\text{NH}_3$  and  $\text{HCl}$ , must be dissolved in  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ , an excess of  $\text{NH}_3$ ,  $\text{NH}_4\text{Cl}$ , and  $\text{MgSO}_4$  added. Precipitate,  $\text{Mg}(\text{NH}_4)\text{PO}_4$ .

33.  $\text{H}_3\text{PO}_4$  and Cr. The insoluble in  $\text{NaOH}$  (sec. 31) is dissolved in  $\text{HCl}$ , neutralised with  $\text{NH}_3$ , excess of  $(\text{NH}_4)_2\text{S}$  added, and filtered. To precipitate  $\text{H}_3\text{PO}_4$ ,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$ , and  $\text{MgSO}_4$  are added to the filtrate.—To prove the presence of Cr in the black precipitate of  $\text{FeS}$ , the latter, after being dried, is fused with a mixture of  $\text{Na}_2\text{CO}_3$ , and  $\text{KNO}_3$  or  $\text{KClO}_3$ , in a porcelain crucible. The melted mass is dissolved in water, filtered, and the chromic acid precipitated in the filtrate by addition of  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ , as  $\text{PbCrO}_4$ .—Or proceed as directed in sec. 19.

34. Mn, Zn, Ba, Sr, Ca, Mg. The filtrate from the precipitate with  $\text{FeCl}_3$  and  $\text{HC}_2\text{H}_3\text{O}_2$  is treated with  $\text{NH}_3$ ,  $\text{NH}_4\text{Cl}$ , and  $(\text{NH}_4)_2\text{S}$ . Precipitate:  $\text{ZnS}$  and  $\text{MnS}$ . This precipitate is dissolved in  $\text{HCl}$ , and investigated as in sec. 14. In the filtrate from  $\text{ZnS}$ , and  $\text{MnS}$ , the presence of Ba, Sr, Ca, and Mg is proved as in sec. 4.

35. *Additional Remarks.*—Cr and  $\text{H}_3\text{PO}_4$ . In separating phosphoric acid from Ba, Sr, Ca, and Mg, as in sec. 31, the chromium is sometimes not thoroughly precipitated, and a coloured filtrate is obtained. The separation by means of  $\text{BaCO}_3$  is more exact, but of course the presence of Ba must be tested for. After it has been ascertained, as in sec. 29, 30, that Fe and  $\text{H}_3\text{PO}_4$  are present in the  $\text{HCl}$  solution of the  $(\text{NH}_4)_2\text{S}$  precipitate,  $(\text{NH}_4)_2\text{SO}_4$  is added to another portion of the solution. The precipitate of  $\text{BaSO}_4$  and  $\text{SrSO}_4$  is investigated as in sec. 6. (Or Ba and Sr may be tested for with lime-water.) To the remainder of the solution add  $\text{FeCl}_3$  in case iron be not present in sufficient quantity, till a drop of the solution gives a yellow precipitate when  $\text{NH}_3$  is added. The liquid is now evaporated *nearly* to dryness, the residue dissolved in  $\text{H}_2\text{O}$ , the excess of acid nearly neutralised with  $\text{Na}_2\text{CO}_3$ , and  $\text{BaCO}_3$  added; the mixture is shaken, and allowed to stand in the cold. The precipitate is dissolved in  $\text{HCl}$ , the Ba removed by  $\text{H}_2\text{SO}_4$  and  $\text{NH}_3$  added. Precipitate,

$\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{H}_3\text{PO}_4$ . The filtrate from the precipitate with  $\text{BaCO}_3$  is treated with  $\text{H}_2\text{SO}_4$ , to precipitate the barium in solution, and proceeded with as in sec. 34.

### GROUPS V. and VI. †

36. The solution of the substance if aqueous is acidified with  $\text{HCl}$ ; if acid, diluted with water, and without paying regard to the precipitate\* which may have been formed, a solution of  $\text{H}_2\text{S}$  is added. If a precipitate falls, the fluid must be gently warmed, and  $\text{H}_2\text{S}$  passed through till it smells of it even after being shaken, and a portion filtered does not give a precipitate.

If the solution of substance contains too much free  $\text{HNO}_3$ , only a flocky yellow precipitate of sulphur is formed on treating with  $\text{H}_2\text{S}$ ; nitrous fumes are also evolved. The acid must be expelled by evaporation, and the solution sufficiently diluted.

*All the sulphides of metals belonging to groups V. and VI. are coloured.*—If a white precipitate fall or only a milky turbidity of S, it may be concluded that groups V. and VI. are absent; also that no  $\text{Cr}_2\text{O}_3$ , or  $\text{Fe}_2\text{O}_3$  is present. If the chromium be contained in the solution, the reddish-yellow colour is changed to violet or green. Take no further notice of the precipitate of S, but saturate the fluid, without submitting it to filtration, with

\* On adding water to an acid solution, basic bismuth and antimony salts are thrown down.— $\text{HCl}$  precipitates  $\text{AgCl}$ ,  $\text{PbCl}_2$ , and  $\text{Hg}_2\text{Cl}_2$ .— $\text{HCl}$  added to an alkaline solution may precipitate  $\text{AgCl}$ ,  $\text{PbCl}_2$ ,  $\text{As}_2\text{S}_3$ ,  $\text{SnS}_2$  (e.g.,  $\text{PbO}$  dissolved in  $\text{NaOH}$ ; ammoniacal solution of  $\text{AgCl}$ ,  $\text{As}_2\text{S}_3$ , etc.); and if to the aqueous solution of  $\text{Na}_3\text{SbS}_4$  (Sodic-sulphantimoniate)  $\text{Sb}_2\text{S}_3$  is thrown down, with simultaneous evolution of  $\text{H}_2\text{S}$ . Added to solutions of the double cyanides,  $\text{HCl}$  precipitates insoluble cyanides, with evolution of  $\text{HCN}$ . These precipitates might be filtered off, and investigated alone; but as they are changed into sulphides, that treatment with  $\text{H}_2\text{S}$  is unnecessary.  $\text{HCl}$  produces a precipitate of silica when added to alkaline silicates. For analysis of silicates, see sec. 82.—Cyanides are to be treated as given in sec. 87.

$\text{NH}_3$ , and test for metals of groups III. and IV. with  $(\text{NH}_4)_2\text{S}$ .

37. The precipitate of the sulphides is filtered off, washed, and a small portion gently warmed in a test-tube, with  $(\text{NH}_4)_2\text{S}$ . If a residue remain (group V.), it is filtered off, and excess of  $\text{HCl}$  added to the filtrate. If a *coloured* precipitate be produced, metals belonging to group VI. are present (sec. 43), and the whole precipitate must be treated in the same manner. If, however, the precipitate be pure white (S), metals of the sixth group are not present, and the remainder of the  $\text{H}_2\text{S}$  precipitate may be treated at once with  $\text{HNO}_3$  as given in sec. 38.

### Group V.—Hg, Ag, Pb, Bi, Cu, Cd.

38. The portion of the precipitate, formed by  $\text{H}_2\text{S}$ , which remained insoluble, after being digested with  $(\text{NH}_4)_2\text{S}$ , is washed with  $(\text{NH}_4)_2\text{S}$  water, and boiled with dilute  $\text{HNO}_3$ . Residue:  $\text{HgS}$  (sec. 39); filtrate (sec. 40).

39. Hg. If the residue is heavy and black, it probably consists of  $\text{HgS}$ ; if white or yellow, it is merely sulphur, produced by the oxidation of the sulphides. If the precipitate by  $\text{H}_2\text{S}$  has not been treated with a sufficient quantity of  $(\text{NH}_4)_2\text{S}$ , if  $\text{Hg}_2\text{O}$  was present,  $\text{Hg}_2\text{S}$  remains behind, which is only partially soluble in  $\text{HNO}_3$ , and is partly changed into white or grey  $\text{Hg}(\text{NO}_3)_2 \cdot 2\text{HgS}$ . In order to test for Hg in this residue, it is ignited with dry sodic carbonate (XXV. 11). A similar precipitate may be formed, in presence of  $\text{HgO}$ , if not enough of  $\text{H}_2\text{S}$  has been added in precipitation, and too small a quantity used of  $(\text{NH}_4)_2\text{S}$  in washing.

To confirm the presence of Hg in the black residue, it is dried and heated with  $\text{NaCO}_3$  in a small tube. It may also be confirmed by boiling with  $\text{HNO}_3$  till only a small residue of S remains. The acid solution is then neutralised with  $\text{NaOH}$ , and a solution of KI

added. Red precipitate:  $\text{HgI}_2$ .—Or the  $\text{HgS}$  may be oxidised with a mixture of  $\text{HCl}$  and a small quantity of  $\text{KClO}_3$ ; the excess of acid removed by evaporation, and a few drops of  $\text{SnCl}_2$  added. Precipitate of  $\text{Hg}_2\text{Cl}_2$ .

*Additional Remark.*— $\text{Hg}_2\text{O}$  and  $\text{HgO}$ . If the original substance gives, on addition of  $\text{HCl}$ , a white precipitate ( $\text{Hg}_2\text{Cl}_2$ ) turning black on being treated with  $\text{NaOH}$ , a *mercurous* salt is present. If a *mercuric* salt be also present, it may be recognised in the filtrate from  $\text{Hg}_2\text{Cl}_2$ , by forming a further precipitate of  $\text{Hg}_2\text{Cl}_2$ , on addition of  $\text{SnCl}_2$ . It must be noticed that, by dissolving in  $\text{HNO}_3$ , or in presence of reducing agents, *mercurous* ( $\text{Hg}_2\text{O}$ ) are changed to *mercuric* salts ( $\text{HgO}$ ).

40. **Pb and Ag.** The nitric acid solution of the remaining metals is treated with dilute  $\text{H}_2\text{SO}_4$ .—Precipitate,  $\text{PbSO}_4$ . This precipitate gives a metallic bead on charcoal before the blowpipe; it is soluble in ammoniac tartrate, from which it may be again precipitated by  $\text{K}_2\text{Cr}_2\text{O}_7$ .—Add some  $\text{HCl}$  to the filtrate from the  $\text{PbSO}_4$ . Precipitate,  $\text{AgCl}$ . The precipitate must be easily soluble in  $\text{NH}_3$ ; and, on addition of  $\text{HNO}_3$ , must be again thrown down.

41. To the filtrate from  $\text{AgCl}$ , excess of  $\text{NH}_3$  is added. Precipitate: **Bi**. Dissolve the precipitate, after washing it well in a small quantity of strong  $\text{HCl}$ , and add some water. Precipitate:  $\text{BiOCl}$ .

42. **Cu and Cd.** If the filtrate from the  $\text{Bi}(\text{OH})_3$  has a blue colour, **Cu** is present. To detect **Cd**,  $\text{H}_2\text{S}$  is passed through the fluid, and the precipitate, after being well washed, is boiled in weak  $\text{H}_2\text{SO}_4$  (one part  $\text{H}_2\text{SO}_4$  to five parts  $\text{H}_2\text{O}$ ), with addition of a small quantity of  $\text{H}_2\text{S}$  water. Residue:  $\text{CuS}$ ; solution **Cd**. Test for **Cd** in the filtrate with  $\text{H}_2\text{S}$ . Another method.—Add  $\text{KCN}$  to the ammoniacal solution of **Cu** and **Cd**, till it becomes colourless; and add  $\text{H}_2\text{S}$ . Yellow precipitate,  $\text{CdS}$  ( $\text{CuS}$  is soluble in  $\text{KCN}$ ). Confirm the **Cd** before the blowpipe (XXIV. 1). If **Cu** be absent, **Cd** is tested for at once with  $(\text{NH}_4)_2\text{S}$ .

## Group VI.—As, Sb, Sn.

43. HCl is added to the solution of the metals of group VI. in  $(\text{NH}_4)_2\text{S}$  till it has an acid reaction. If they are absent, a white milky turbidity, produced by the decomposition of *yellow*  $(\text{NH}_4)_2\text{S}$ , will be formed; but if present, a coloured precipitate is thrown down; the solution, in the latter case, is slightly warmed, filtered, and well washed. The mixture of the sulphides is heated gently with concentrated HCl. Yellow residue: As (sec. 44). Filtrate: Sn and Sb (sec. 47).

44. As. The yellow  $\text{As}_2\text{S}_5^*$  is dissolved in HCl, some  $\text{KClO}_3$  being added; excess of  $\text{NH}_3$  is added to the solution, and then  $\text{NH}_4\text{Cl}$ , and  $\text{MgSO}_4$ . *Precipitate:*  $\text{MgNH}_4\text{AsO}_4$ . In order to confirm this, filter off a part of the precipitate, and treat it, after having washed it, with a solution of  $\text{AgNO}_3$ . It assumes a reddish-brown colour:  $\text{Ag}_3\text{AsO}_4$ . The remainder of the precipitate is ignited with KCN, and  $\text{Na}_2\text{CO}_3$  (sec. 45).

45. The presence of As<sup>†</sup> may also be confirmed, by heating the dry  $\text{As}_2\text{S}_5$  mixed with about six times its weight of equal parts of dry  $\text{Na}_2\text{CO}_3$  and KCN, in a glass tube. It is warmed gently at first to expel any moisture which may be present, which is removed from the sides of the tube, where it has condensed, by filter paper, and then heated more strongly. Black metallic deposit: As. If the tube be cut off, and heated, while held in an inclined position, the smell of garlic may be distinctly observed.  $\text{MgNH}_4\text{AsO}_4$  may be reduced in the same manner.

\* As is always precipitated by  $\text{H}_2\text{S}$  from acid solutions, as  $\text{As}_2\text{S}_3$  (XXIV. 10). But when treated with *yellow*  $(\text{NH}_4)_2\text{S}$ , it is converted into  $\text{As}_2\text{S}_5$ .

† When  $\text{As}_2\text{S}_5$  is treated with KCN and  $\text{Na}_2\text{CO}_3$ , the whole of the As is not liberated in the metallic state; for a potassic arsenic sulphide is formed, on which the excess of KCN is without action. If, as is usually the case, free S be present, still less As is reduced. The arsenic sulphide should, in this case, be dissolved by treating with  $\text{NH}_3$ ; the S remains insoluble. The ammoniacal solution is evaporated to dryness, and the residue ignited with KCN and  $\text{Na}_2\text{CO}_3$ .

46. *Additional Remark.*— $\text{As}_2\text{O}_3$  and  $\text{As}_2\text{O}_5$ . Only the alkali-salts of these acids are soluble in water; all other salts dissolve only in acids.  $\text{As}_2\text{O}_3$  may be distinguished from  $\text{As}_2\text{O}_5$  by its behaviour when treated with  $\text{H}_2\text{S}$  (XXIX. 7, 10); *only*  $\text{As}_2\text{O}_5$  gives a precipitate with  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$ , and  $\text{MgSO}_4$ ; the reaction when treated with  $\text{AgNO}_3$  is also different (XXIX. 9, 11). Both argentic arsenite and arseniate are soluble in  $\text{NH}_3$ , but only the solution of argentic arsenite deposits metallic silver when boiled.—A solution of  $\text{As}_2\text{O}_3$  in excess of  $\text{NaOH}$ , when treated with  $\text{CuSO}_4$ , gives a blue solution, from which a red precipitate of  $\text{Cu}_2\text{O}$  is thrown down on boiling. ( $\text{As}_2\text{O}_3 + 6\text{NaOH} + 4\text{CuO} = 2\text{Cu}_2\text{O} + 2\text{Na}_3\text{AsO}_4 + 3\text{H}_2\text{O}$ ). It differs on this account from  $\text{As}_2\text{O}_5$ .

47. **Sn and Sb.**—The acid solution is diluted (if much free acid be present, it must be nearly neutralised with  $\text{NaOH}$ ), and a piece of metallic zinc added. If a small portion of the fluid be reduced with  $\text{Zn}$  on a platinum lid, the latter becomes covered with a black stain of metallic  $\text{Sb}$ . (XXVIII. 9). If the reduction is complete, the excess of  $\text{Zn}$  is removed, the solution of  $\text{ZnCl}_2$  poured off, the deposit washed by decantation, and finally treated with  $\text{HCl}$ . Residue:  $\text{Sb}$ ; Solution— $\text{Sn}$ . The presence of  $\text{Sn}$  in the solution is confirmed with  $\text{HgCl}_2$  (XXV. 10), or with  $\text{H}_2\text{S}$  (brown precipitate,  $\text{SnS}$ , XXVII. 7); if the former of these tests be employed, some  $\text{H}_2\text{SO}_4$  must first be added, to remove any  $\text{Pb}$  which may have been present in the  $\text{Zn}$ . The residue of  $\text{Sb}$  is dissolved in  $\text{HNO}_3$ ,  $\text{HCl}$  added to the solution, and the fluid diluted;  $\text{SbOCl}$  is precipitated, which is soluble in  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ , and precipitable from the solution by  $\text{H}_2\text{S}$ ; the residue may also be confirmed by passing  $\text{H}_2\text{S}$  through the nitric acid solution; but the presence of  $\text{Pb}$  renders the action indistinct.

48. **Oxides of Sn and Sb.**—A solution of a stannous salt may be recognised by its behaviour, when treated with  $\text{HgCl}_2$ ;  $\text{SnO}_2$  may be precipitated from a *weak*



acid solution, when poured into a saturated solution of  $\text{Na}_2\text{SO}_4$  ( $\text{SnO}_2$ ). A solution of  $\text{Sb}_2\text{O}_5$ , when boiled with  $\text{KI}$  and  $\text{HCl}$ , liberates free iodine. ( $\text{Sb}_2\text{O}_5 + 4\text{KI} + 10\text{HCl} = 4\text{I} + 4\text{KCl} + 2\text{SbCl}_3 + 5\text{H}_2\text{O}$ ).  $\text{AgNO}_3$ , added to a solution of  $\text{Sb}_2\text{O}_3$  in  $\text{NaOH}$  gives a black precipitate of a mixture of  $\text{Ag}_4\text{O}$  and  $\text{Ag}_2\text{O}$ . If  $\text{NH}_3$  be added,  $\text{Ag}_2\text{O}$  dissolves, and a deep black residue of  $\text{Ag}_4\text{O}$  remains. ( $\text{Sb}_2\text{O}_3 + 8\text{AgNO}_3 + 8\text{NaOH} = 2\text{Ag}_4\text{O} + \text{Sb}_2\text{O}_3 + 8\text{NaNO}_3 + 4\text{H}_2\text{O}$ ).

### Second Process for Separating As, Sb, Sn.

49. The mixture of sulphides VI. (sec. 43) is heated with concentrated  $\text{HCl}$ , to which some  $\text{KClO}_3$  has been added; at first gently, then more strongly, till nothing but  $\text{S}$  remains undissolved. The solution is filtered, excess of pretty concentrated  $\text{NaOH}$  is added, and then a quantity of alcohol, equal in volume to a fifth of the solution. Precipitate:  $\text{NaSbO}_3$ . Confirm before the blowpipe with  $\text{Na}_2\text{CO}_3$ ; or by dissolving in  $\text{HCl}$ , and depositing the  $\text{Sb}$  on platinum with  $\text{Zn}$ . The filtrate is evaporated till the alcohol is removed; it is then acidified with  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ , to prevent the precipitation of  $\text{SnO}_2$ , and excess of  $\text{NH}_3$  added. The  $\text{As}$  is now precipitated as  $\text{MgNH}_4\text{AsO}_4$  by addition of  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$ , and  $\text{MgSO}_4$ . (Confirm as in sec. 44). The filtrate from the ammonio-magnesian-arsenate is acidified with  $\text{HCl}$ , and  $\text{H}_2\text{S}$  added. Precipitate:  $\text{Sn}$ . Confirm by igniting the precipitate in the oxidising flame, and afterwards reducing the resulting  $\text{SnO}_2$  with  $\text{Na}_2\text{CO}_3$  on charcoal before the blowpipe.

### Third Method of Separation of As, Sb, and Sn.

50. The sulphides VI. (sec. 43) are dissolved in  $\text{Na}_2\text{S}$  (prepared by adding an excess of  $\text{H}_2\text{S}$  to a solution of  $\text{NaOH}$ , and then adding an equal volume of the same solution of  $\text{NaOH}$ ), an excess of  $\text{SO}_2$  added, and the solution heated gently at first, and then boiled till all excess

of  $\text{SO}_2$  is expelled. Precipitate: Sn and Sb ; solution : As. —( $\text{As}_2\text{S}_3 + 3\text{Na}_2\text{S} + 5\text{S}_2 = 2\text{NaAsO}_2 + 2\text{Na}_2\text{S}_2\text{O}_3 + \text{S}_7$ ). The residue of the sulphides of Sn and Sb is dissolved in HCl, and Sn separated from Sb, as in sec. 47, or sec. 49. In the filtrate, As is precipitated as  $\text{As}_2\text{S}_3$  on addition of  $\text{H}_2\text{S}$ .

### Analysis of Compounds Insoluble in Water and Acids.

51. To this class belong, 1st,  $\text{BaSO}_4$ ,  $\text{SrSO}_4$ ,  $\text{CaSO}_4$ ,  $\text{PbSO}_4$ ;—2nd, AgCl, AgBr, AgI,  $\text{PbCl}_2$ ;—3rd, Oxides which have been ignited, such as  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$ , etc.;—4th,  $\text{CaFl}_2$ ;—5th, C, S;—6th, Silicates.

52. If the substance be insoluble and black, it should be ignited on platinum foil in the oxidising flame of the blowpipe. If C be present it is oxidised, and the residue becomes white. At the same time S, if it be present, may be discovered by the odour of  $\text{SO}_2$ ; it may also be recognised by its subliming in brown drops when heated in a glass tube (I. 2).

53. A portion of the substance, after C and S have been removed, is treated with  $(\text{NH}_4)_2\text{S}$ . If it becomes black, Pb or Ag are present, and may be confirmed by the blowpipe tests.  $\text{SnO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{SO}_3$ ,  $\text{SiO}_2$  may also be discovered by the blowpipe— $\text{CaFl}_2$ , *vide* sec. 64, p. 59. Analysis of silicates, *vide* sec. 82, p. 63.

54. If Pb and Ag be absent, the insoluble substance is fused with four times its weight of  $\text{Na}_2\text{CO}_3$  in a platinum crucible, with the foot-blowpipe, till the mass becomes fluid. On treating with water, the *acids* dissolve and the bases remain behind ( $\text{BaO}$ ,  $\text{SrO}$ , etc.); they are carefully washed out with water, dissolved in HCl, and separated as usual. If chromium be present, as soon as the mixture is fluid,  $\text{KClO}_3$  is added in small portions at a time, or the mixture may be fused with equal parts of  $\text{Na}_2\text{CO}_3$ , and  $\text{KNO}_3$  in a porcelain crucible, for ten minutes, taking care not to use too great heat. In the aqueous solution

of the fused mass, the  $\text{CrO}_3$  may be recognised by acidifying with  $\text{HCl}$ , and precipitation with  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ . Ignited  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  are soluble by continued heating with a mixture of equal volumes of concentrated  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{O}$ .—Ignited  $\text{SnO}_2$  is not altered by fusion with  $\text{Na}_2\text{CO}_3$ . It must be fused with six times its weight of a mixture of equal parts of  $\text{Na}_2\text{CO}_3$  and  $\text{S}$  in a porcelain crucible. The fused mass is dissolved in water and  $\text{SnS}_2$  precipitated on addition of  $\text{HCl}$ , which is further investigated as in sec. 43.

55. *Ag and Pb are present.*—*a.* The substance is repeatedly boiled with a concentrated solution of  $\text{NH}_4(\text{C}_2\text{H}_3\text{O}_2)$  which extracts  $\text{PbSO}_4$  and  $\text{PbCl}_2$ . The  $\text{Pb}$  may be recognised in the filtrate by  $\text{H}_2\text{S}$ ; the  $\text{Cl}$ , by  $\text{AgNO}_3$ ; the  $\text{H}_2\text{SO}_4$  by means of  $\text{BaCl}_2$ . The residue, insoluble in  $\text{NH}_4(\text{C}_2\text{H}_3\text{O}_2)$  is treated with  $\text{NH}_3$  by which  $\text{AgCl}$  is extracted; its presence in the filtrate may be confirmed by addition of  $\text{HNO}_3$ .  $\text{AgBr}$ , and  $\text{AgI}$  are, however, almost insoluble in  $\text{NH}_3$ ; the solution is therefore treated with an aqueous solution of  $\text{KCN}$ . By acidulating the solution in  $\text{KCN}$  with  $\text{HNO}_3$ , the dissolved  $\text{Ag}$  is precipitated. The united precipitate of  $\text{AgCl}$ ,  $\text{AgBr}$ , and  $\text{AgI}$  is fused in a porcelain crucible with  $\text{Na}_2\text{CO}_3$ , and in the aqueous extract,  $\text{Cl}$ ,  $\text{Br}$ , and  $\text{I}$  are tested for. It may, instead, be treated with  $\text{Zn}$ , and very dilute  $\text{H}_2\text{SO}_4$  (sec. 63).

The residue, from which  $\text{Ag}$  and  $\text{Pb}$  have been removed, is investigated as in sec. 54.

56. *b.* The substance, insoluble in acids, is treated with  $\text{NaHCO}_3$  (were  $\text{Na}_2\text{CO}_3$  used, some  $\text{Pb}$  would be dissolved), and allowed to stand for some hours in the cold. It is then filtered, washed with cold water, and  $\text{HCl}$ , and  $\text{H}_2\text{SO}_4$ , tested for in the filtrate as usual. The residue is treated with dilute  $\text{HNO}_3$ , when  $\text{Pb}$ ,  $\text{Ca}$ , and  $\text{Sr}$  are dissolved, while  $\text{BaSO}_4$ ,  $\text{AgCl}$ ,  $\text{AgBr}$ , and  $\text{AgI}$  are left insoluble. The insoluble residue is fused with  $\text{Na}_2\text{CO}_3$  in a porcelain crucible, and the fused mass treated repeatedly with water;  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{HBr}$ , and  $\text{HI}$  are dissolved,

while  $\text{BaCO}_3$  and  $\text{AgCl}$  remain behind, and may be separated by  $\text{HCl}$ . The presence of  $\text{BaSO}_4$  may also be recognised in the original mixture, by repeated boiling with fresh portions of  $\text{Na}_2\text{CO}_3$  (XXXVI. 3, p. 31); or the silver salts may be extracted with  $\text{KCN}$  and the residue of  $\text{BaSO}_4$  decomposed with  $\text{Na}_2\text{CO}_3$ .

### C.—DETECTION OF ACIDS.

57. The following substances are soluble in *water*:—

1. *All salts* of the following acids;  $\text{HNO}_3$ ,  $\text{HClO}_3$ ,  $\text{HNO}_2$ ,  $\text{H}_2\text{SO}_4$  (except  $\text{BaSO}_4$ ,  $\text{SrSO}_4$ ,  $\text{CaSO}_4$ ,  $\text{PbSO}_4$ ),  $\text{HCl}$  (except  $\text{AgCl}$ ,  $\text{Hg}_2\text{Cl}_2$ ,  $\text{PbCl}_2$ ),  $\text{HBr}$  (except  $\text{AgBr}$ ,  $\text{Hg}_2\text{Br}_2$ ),  $\text{HI}$  (except  $\text{AgI}$ ,  $\text{HgI}_2$ ,  $\text{Hg}_2\text{I}_2$ ,  $\text{Cu}_2\text{I}_2$ ),  $\text{HCHO}_2$  (formic acid),  $\text{HC}_2\text{H}_3\text{O}_2$ .

2. The *alkali-salts* of the following anhydrides:— $\text{CO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{As}_2\text{O}_5$ ,  $\text{P}_2\text{O}_5$ ,  $\text{SO}_2$ ,  $\text{SiO}_2$ ,  $\text{Bo}_2\text{O}_3$ ; and of the acid  $\text{H}_2\text{C}_2\text{O}_4$ . All other salts, with the exception of silicates, are soluble in acids.

3. The *sulphides* and *cyanides* of the alkalis.

$\text{CO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{SO}_2$ ,  $\text{As}_2\text{O}_3$ ,  $\text{As}_2\text{O}_5$ , and  $\text{CrO}_3$  may be discovered in the usual course of analysis;  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{SO}_2$  are evolved as gases, on treating their salts with  $\text{HCl}$ .  $\text{CO}_2$  renders lime water turbid;  $\text{H}_2\text{S}$  blackens paper moistened with a solution of  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)$ ; and  $\text{SO}_2$  has the same effect on paper, which has been dipped into a solution of  $\text{Hg}_2(\text{NO}_3)_2$ .

#### Detection of $\text{HCl}$ , $\text{HNO}_3$ , $\text{H}_2\text{SO}_4$ , $\text{H}_3\text{PO}_4$ .

58.  $\text{H}_2\text{SO}_4$ . The aqueous solution of the substance is acidified with  $\text{HCl}$ , and  $\text{BaCl}_2$  added. If the solution is acid already, no more need be added. White precipitate;  $\text{BaSO}_4$ . If  $\text{Pb}$ ,  $\text{Ag}$ , or  $\text{Hg}_2\text{O}$  be present, the solution must be acidified with  $\text{HNO}_3$ , and  $\text{Ba}(\text{NO}_3)_2$  added.

$\text{HCl}$ . The *aqueous* solution is acidified with  $\text{HNO}_3$ , and  $\text{AgNO}_3$  added. The white, curdy precipitate must be completely soluble in  $\text{NH}_3$ .

$\text{HNO}_3$ . A few drops of a solution of  $\text{FeSO}_4$  are mixed

with concentrated  $\text{H}_2\text{SO}_4$ ; the mixture is *thoroughly* cooled, and the aqueous solution of the substance poured on to the top of the denser fluid. Reddish-brown ring at the point of contact:  $\text{HNO}_3$  (*vide* IV. 6, p. 11).

$\text{H}_3\text{PO}_4$ . As  $\text{H}_3\text{PO}_4$  is not to be distinguished from  $\text{H}_3\text{AsO}_4$  by the following tests, if present, the latter must be removed by means of  $\text{H}_2\text{S}$ .

59. *a.* A substance, soluble in water, can contain only alkali-salts of  $\text{H}_3\text{PO}_4$ . To the aqueous solution,  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_3$ , and  $\text{MgSO}_4$  are added. Precipitate:  $\text{Mg}(\text{NH}_4)\text{PO}_4$ .—If the solution contain  $\text{CO}_2$ , it must first be removed by  $\text{HCl}$ , else  $\text{MgCO}_3$  might be precipitated. The acidified solution is boiled, and treated as before.

*b.* If the substance be soluble only in acids,  $\text{NH}_3$  is added to the solution till a precipitate is formed. It is dissolved in a small quantity of  $\text{HC}_2\text{H}_3\text{O}_2$ , some  $\text{NaC}_2\text{H}_3\text{O}_2$  is added, and then *one* drop of  $\text{FeCl}_3$ . Yellowish-white, gelatinous precipitate:  $\text{FePO}_4$ .

Another method:—The solution of the substance—best in  $\text{HNO}_3$ —is treated as in sec. 30, p. 46. The precipitate of ammoniac phospho-molybdate is dissolved in  $\text{NH}_3$ ,  $\text{NH}_4\text{Cl}$  and  $\text{MgSO}_4$  added, and the phosphoric acid precipitated as  $\text{Mg}(\text{NH}_4)\text{PO}_4$ . The precipitate of  $\text{Mg}(\text{NH}_4)\text{PO}_4$  should, when filtered off, acquire a yellow colour, when treated with  $\text{AgNO}_3$  ( $\text{Ag}_3\text{PO}_4$ ).

### Detection of Other Acids.

60. Some of the solid substance in a test-tube is moistened with concentrated  $\text{H}_2\text{SO}_4$ . If an immediate reaction is not observed, heat gently.

1. *Colourless gas escapes:*  $\text{HCl}$ ,  $\text{HNO}_3$ ,  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{SO}_2$  (from sulphites and hypo-sulphites),  $\text{HFl}$  (the vapours attack the glass),  $\text{CO}$  (by decomposition of *cyanides* or *organic acids*).

2. *Gas which has a colour escapes:* Brownish-yellow:  $\text{Br}$ ;—red:  $\text{HNO}_2$ ;—violet:  $\text{I}$ ;—greenish-yellow:  $\text{HClO}$  and  $\text{HClO}_2$ .

3. *Acids which are not volatile*:  $\text{H}_3\text{PO}_4$ ,  $\text{SiO}_2$ ,  $\text{B}_2\text{O}_3$  (*vide* VIII. 6, p. 13).

4. *The substance turns black*: Organic acids.

61. **HI** and **HBr**. In order to detect I, the aqueous solution of the substance is acidified with  $\text{H}_2\text{SO}_4$ , some boiled starch, and a drop of  $\text{NO}_2$  are added. If iodine be present, the solution assumes a blue colour. Instead of using starch, some  $\text{CS}_2$ , or benzole, may be added, and the mixture, after addition of  $\text{KNO}_2$ , well shaken. If the  $\text{CS}_2$ , or benzole, have, on settling, a rose colour, I is present.—To detect Br,  $\text{CS}_2$  is added to the solution, and then chlorine water, or solution of  $\text{NaClO}$  drop by drop. Shake after each addition of chlorine, and notice if the  $\text{CS}_2$  has a yellow colour; if such be the case Br is present. If iodine be present at the same time, the rose colour is first seen. Chlorine water is added till the rose colour disappears. If a drop more be added, the yellow colour, produced by bromine, will become apparent.

62. **Cl**, **Br**, and **I**.—I and Br are first tested for as in sec. 61. In order to test for Cl, the solution, which must only contain alkaline metals (if others be present, they must be removed by boiling, or fusing with excess of  $\text{Na}_2\text{CO}_3$ ) is treated with  $\text{Na}_2\text{CO}_3$ , and  $\text{K}_2\text{Cr}_2\text{O}_7$ , evaporated to dryness, and the residue fused in a porcelain crucible. If iodine be present, the vapours will here escape. The melted mass is poured out, broken into small pieces, and heated in a small retort with an excess of very concentrated  $\text{H}_2\text{SO}_4$ . Reddish-brown drops distil over ( $\text{CrO}_2\text{Cl}_2$ , as well as Br and I). Water is carefully added, and the solution tested for Cr, by boiling with alcohol, and precipitating with  $\text{NH}_3$  (XV. 9, 11, p. 17). If Cr is found, Cl was present in the original substance.

63. **AgCl**, **AgBr**, **AgI**.—**AgBr** has a light yellow colour, and is but slightly soluble in  $\text{NH}_3$ ; **AgI** is yellow and almost insoluble in  $\text{NH}_3$ . If a solution, containing

Cl, Br, and I, be treated with excess of  $\text{AgNO}_3$ ,  $\text{NH}_3$  added to the precipitate, dissolves only  $\text{AgCl}$ , with a small quantity of  $\text{AgBr}$ . The ammoniacal solution, when treated with  $\text{HNO}_3$  gives a precipitate of  $\text{AgCl}$ , with which a little  $\text{AgBr}$  is mixed. When  $\text{AgI}$  is treated with  $\text{NH}_3$  it grows white, on account of the absorption of  $\text{NH}_3$ .

To recognise  $\text{AgCl}$ ,  $\text{AgBr}$ , or  $\text{AgI}$ , in presence of each other, the mixture, previously fused, is moistened with dilute  $\text{H}_2\text{SO}_4$ , and a small piece of  $\text{Zn}$  laid on the top. It is allowed to stand for 24 hours; the solution is then poured off, the residue well washed with  $\text{H}_2\text{O}$ , and the filtrate boiled with excess of  $\text{Na}_2\text{CO}_3$ . The fluid is filtered from the precipitate of  $\text{ZnCO}_3$ ; the solution contains  $\text{NaCl}$ ,  $\text{NaBr}$ ,  $\text{NaI}$ , and is treated further as in sec. 62.

64.  $\text{HF}$ .—The substance, reduced to fine powder, is heated with concentrated  $\text{H}_2\text{SO}_4$  in a dry test-tube. The vapours of  $\text{HF}$  make the sides of the glass tube dim. A better method is to form a thick paste, by mixing the powdered substance with concentrated  $\text{H}_2\text{SO}_4$ , and warm it gently in a platinum crucible, covered by a watch glass, which has been coated with wax on its convex side, and some scratches made on it. On to the concave side some water is poured, in order better to condense the vapours. After five or ten minutes, the watch-glass is removed, the most of the wax melted off by rubbing the glass, slightly heated, with filter paper, and thoroughly cleaned with alcohol. If  $\text{HF}$  was present, the glass will have been distinctly etched. See sec. 85, p. 64.

65.  $2\text{HF}$ ,  $\text{SiF}_4$ .—All salts of this acid, with exception of the  $\text{K}$ ,  $\text{Na}$ , and  $\text{Ba}$  salts are soluble in water. When heated with  $\text{H}_2\text{SO}_4$ , a mixture of  $\text{HF}$ , and  $\text{SiF}_4$  escapes, which acts on the glass of the test tube. When heated in a test tube the *dry* salts evolve  $\text{SiF}_4$ , which, when led into water, deposits a gelatinous precipitate of  $\text{SiO}_2$  (*vide* sec. 85, p. 64). The fluosilicates, when heated with excess of an alkali, are decomposed into  $\text{SiO}_2$ , and the

alkaline fluoride. If fused with  $\text{Na}_2\text{CO}_3$ ,  $\text{NaFl}$ , and sodic silicate is formed. If heated with microcosmic salt, before the blowpipe, they give no skeleton, for all Si volatilises as  $\text{SiFl}_4$ .

66.  $\text{SO}_2$ , and  $\text{H}_2\text{S}_2\text{O}_3$ . The salts of both of these acids, when heated with  $\text{H}_2\text{SO}_4$  evolve  $\text{SO}_2$  which may be recognised by its smell, and by its reaction on paper moistened with a solution of  $\text{Hg}_2(\text{NO}_3)_2$ , which is blackened by liberation of metallic mercury. The reaction is, of course, possible, only when  $\text{H}_2\text{S}$  is absent.

$\text{SO}_2$ . The aqueous solution of an alkaline sulphite is made weakly acid with  $\text{HC}_2\text{H}_3\text{O}_2$ , some  $\text{ZnSO}_4$ , and sodic nitro-prusside. The fluid assumes a red colour. If  $4\text{KCN}$ ,  $\text{FeCN}_2$  be present, the colouration is more distinct; if a large quantity of  $\text{SO}_2$  be present, a purple coloured precipitate is formed. To detect small quantities of  $\text{SO}_2$ , or  $\text{H}_2\text{S}_2\text{O}_3$ , metallic Al, or pure Zn is treated with  $\text{HCl}$ , or  $\text{H}_2\text{SO}_4$ , and the substance added.  $\text{H}_2\text{S}$  is evolved, which may be recognised by its behaviour to paper moistened with  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$ .

$\text{H}_2\text{S}_2\text{O}_3$ . The salts of this acid, with exception of  $\text{BaS}_2\text{O}_3$ , and  $\text{PbS}_2\text{O}_3$  are soluble in water. The behaviour of salts of  $\text{H}_2\text{S}_2\text{O}_3$ , with regard to  $\text{HCl}$ , and  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  is characteristic (*vide* XXXIII. 2, 3, p. 29).

$\text{H}_2\text{S}$ ,  $\text{H}_2\text{S}_2\text{O}_3$ , and  $\text{SO}_2$ . If salts of these acids are mixed, and it is required to detect the acids,  $\text{H}_2\text{S}$  is expelled first, and  $\text{ZnSO}_4$ . In a part of the filtrate from the  $\text{ZnS}$ , the hyposulphurous acid is detected, by the solution giving a precipitate of S, when acidified with  $\text{HCl}$ . Another portion is tested for  $\text{SO}_2$  with sodic nitro-prusside.

67.  $\text{HClO}_3$ . The presence of chlorates may be discovered by their behaviour on being heated; by the reaction with concentrated  $\text{H}_2\text{SO}_4$ , and by the characteristic deportment, when treated with indigo and  $\text{SO}_2$  (*vide* XXX. p. 28).

$\text{HClO}_3$  and  $\text{HNO}_3$ .  $\text{HClO}_3$  may be recognised in presence of  $\text{HNO}_3$  by the reaction with indigo and  $\text{SO}_2$ . To confirm  $\text{HNO}_3$ , the substance is treated with excess of



NaOH, and the solution boiled with a mixture of one part iron filings, and two parts Zn.  $\text{NH}_3$  formed by the reduction of the  $\text{HNO}_3$  is evolved.

68.  $\text{HClO}$  (present in "chloride of lime"). The salts have the odour of Cl, and bleach indigo and litmus. If  $\text{MnSO}_4$  be added to a solution of a hypochlorite,  $\text{MnO}_2$  is thrown down. If a dry hypochlorite be heated, Cl is evolved. If a hypochlorite, suspended in water be heated, after addition of a drop of  $\text{Co}(\text{NO}_3)_2$ , O is evolved.

69.  $\text{HNO}_2$ . Nitrites, heated with concentrated  $\text{H}_2\text{SO}_4$ , give off brownish-red vapours ( $\text{N}_2\text{O}_3$ ). With dilute  $\text{H}_2\text{SO}_4$ , only NO is evolved. The aqueous solution of the substance is acidified with  $\text{H}_2\text{SO}_4$ , some starch paste and KI added. Blue colouration;  $\text{HNO}_2$ .—An aqueous solution of  $\text{FeSO}_4$  turns brown, on addition of a nitrous solution, even if *no*  $\text{H}_2\text{SO}_4$  be added. This is a reaction which distinguishes nitrites from nitrates.

70.  $\text{Bo}_2\text{O}_3$ , *vide* VIII. 5, 6, p. 13.

## ORGANIC ACIDS.

The salts of all organic acids, except oxalic acid, blacken, when heated.

71. It is most advantageous in detecting organic acids (as indeed all acids), to have only alkaline salts in solution. Hence, if other metals are present, the substance is boiled with excess of  $\text{Na}_2\text{CO}_3$ . The filtrate, which contains all the acids, after being concentrated by evaporation, is made weakly acid with HCl, to remove excess of  $\text{Na}_2\text{CO}_3$ . It is then gently warmed, to remove the dissolved  $\text{CO}_2$ ;  $\text{NH}_3$  is added till an alkaline reaction is apparent, and then  $\text{CaCl}_2$ . After a lapse of fifteen or twenty minutes, the precipitate of  $\text{CaC}_2\text{O}_4$ , and  $\text{CaC}_4\text{H}_4\text{O}_6$  filtered off (sec. 72). The filtrate is treated as directed in sec. 73.

72. The precipitate of  $\text{CaC}_2\text{O}_4$ , and  $\text{CaC}_4\text{H}_4\text{O}_6$  is filtered off, washed, and treated with NaOH at the usual temperature.  $\text{CaC}_4\text{H}_4\text{O}_6$  dissolves, and when the solution

is filtered off and boiled, reprecipitates.  $\text{CaC}_2\text{O}_4$  remains undissolved.

73. The filtrate from the precipitate of calcic salts is heated for some time, to boiling. Precipitate:  $\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$  (calcic citrate). The precipitate is filtered off while boiling (the filtrate is treated as in sec. 74); it must be soluble in  $\text{HCl}$ , and after saturation with  $\text{NH}_3$ , be again precipitated by boiling.

74. To the filtrate, three times its volume of alcohol is added. The precipitate of calcic succinate ( $\text{CaC}_4\text{H}_4\text{O}_4$ ) is filtered off, and washed with alcohol. The aqueous solution gives a precipitate of ferric succinate, when treated with  $\text{FeCl}_3$ .

75. *Formic and acetic acids.*—If no volatile acids be present, the substance is distilled with dilute  $\text{H}_2\text{SO}_4$ ; the distillate is neutralised with  $\text{Na}_2\text{CO}_3$ , and evaporated to dryness. Part of the residue is heated in a test-tube with dry  $\text{As}_2\text{O}_3$ ; if an extremely unpleasant smell of kakodyl be perceived, acetic acid is present. It may also be tested for by means of alcohol and  $\text{H}_2\text{SO}_4$  (XXXVIII. 4, p. 32). The remainder of the residue is dissolved in  $\text{H}_2\text{O}$ , and boiled with  $\text{AgNO}_3$ . Black precipitate of  $\text{Ag: HCHO}_2$ .

76. *Oxalic acid.*—The aqueous solution of an alkaline oxalate, when acidified with  $\text{HC}_2\text{H}_3\text{O}_2$ , gives, on addition of  $\text{CaCl}_2$ , a white precipitate of  $\text{CaC}_2\text{O}_4$  (*vide* X. 8, p. 14). All other oxalates must be decomposed by boiling with  $\text{Na}_2\text{CO}_3$ , or by  $\text{H}_2\text{S}$ , or  $(\text{NH}_4)_2\text{S}$ , and then tested in the same manner.

77. *Tartaric acid.*—A concentrated *neutral* solution of a tartrate is acidified with  $\text{HC}_2\text{H}_3\text{O}_2$ , and  $\text{KC}_2\text{H}_3\text{O}_2$  added. If the original solution be acid, it need not be further acidified. Precipitate:  $\text{KH.C}_4\text{H}_4\text{O}_6$ .

78. *Citric acid.*— $\text{H}_3\text{C}_6\text{H}_5\text{O}_7$ . If lime-water be added to a solution of free citric acid, till the reaction is alkaline, and then boiled, a precipitate of  $\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$  is thrown down, which dissolves on the solution cooling.

79. *Succinic acid.* —  $\text{H}_2\text{C}_4\text{H}_4\text{O}_4$ ,  $\text{CaC}_4\text{H}_4\text{O}_4$ , and

$\text{BaC}_4\text{H}_4\text{O}_4$  are soluble in  $\text{H}_2\text{O}$ , but not in alcohol.—The brown precipitate which  $\text{FeCl}_3$  produces in the neutral solution of a succinate, is filtered off, washed out, and removed to a test tube; some  $\text{NH}_3$  is added;  $\text{Fe}(\text{OH})_3$  is precipitated; in the filtered solution  $\text{BaCl}_2$ , and a considerable amount of alcohol produce a precipitate of  $\text{BaC}_4\text{H}_4\text{O}_4$ .

80. *Acetic acid*.—Reaction of the salts with  $\text{H}_2\text{SO}_4$  and  $\text{C}_2\text{H}_6\text{O}$  (*vide* XXXVIII. 3, 4, p. 32).—In the neutral solution of the alkaline acetates,  $\text{FeCl}_3$  gives a red colouration, which disappears on boiling, with separation of a brown precipitate.— $\text{AgC}_2\text{H}_3\text{O}_2$  is slightly soluble in cold water; it dissolves more easily in hot water.—If dry  $\text{NaC}_2\text{H}_3\text{O}_2$  be heated with  $\text{As}_2\text{O}_3$ , a penetrating, extremely disagreeable smell of kakodyl may be perceived ( $\text{As}[\text{CH}_3]_2$ ).

81. *Formic acid*.— $\text{H}\cdot\text{CHO}_2$ . Formiates, when heated with dilute  $\text{H}_2\text{SO}_4$ , evolve free formic acid which may be recognised by its smell. When heated with alcohol and concentrated  $\text{H}_2\text{SO}_4$ , formic ether ( $\text{C}_2\text{H}_5\text{CHO}_2$ ) is evolved, which has a smell of arrack.—If a dry formiate be heated,  $\text{CO}$  is evolved.— $\text{AgNO}_3$  added to the aqueous solution of a formiate, produces a white precipitate ( $\text{AgCHO}_2$ ), which, when boiled, deposits metallic silver ( $2\text{AgCHO}_2 = 2\text{Ag} + \text{CO}_2 + \text{HCHO}_2$ ).—If  $\text{NH}_3$  be present, however, no  $\text{Ag}$  is precipitated.—If formic acid or an alkaline formiate be warmed with a solution of mercuric chloride, mercurous chloride (calomel) is thrown down.—With  $\text{FeCl}_3$ ,  $\text{HCHO}_2$  deports itself in the same manner as  $\text{HC}_2\text{H}_3\text{O}_2$ .

$\text{HCHO}_2$  and  $\text{HC}_2\text{H}_3\text{O}_2$ . In a mixture of formic and acetic acids, the latter may be recognised by the kakodyl reaction; the former by its behaviour with  $\text{HgCl}_2$  and  $\text{AgNO}_3$ .— $\text{Pb}(\text{CHO}_2)_2$  is soluble with difficulty in water, and insoluble in alcohol;  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$  is easily soluble in both of these solvents.

## ANALYSIS OF SILICATES.

The presence of  $\text{SiO}_2$  may be recognised in the bead of microcosmic salt, by the preliminary examination (*vide* XXXI. 3, p. 29). Silicates are either only partly decomposed by inorganic acids, or not attacked at all.

82. *Silicates which are decomposable by acids.*—(Water glass, zeolite, slags). The substance, reduced to fine powder, is warmed with concentrated  $\text{HCl}$ , and stirred frequently; it is then diluted with  $\text{H}_2\text{O}$ , and evaporated to dryness. The dry residuc is moistened with concentrated  $\text{HCl}$ , and warmed; water is then added, the fluid is boiled, and the  $\text{SiO}_2$  filtered off. All the bases, formerly existing as silicates, are to be found in the solution as chlorides, and may be separated in the ordinary way. *Pure*  $\text{SiO}_2$  must dissolve in  $\text{Na}_2\text{CO}_3$ , by persistent boiling (*vide* sec. 86).

83. *Silicates which are not attacked by acids.*—*a. Separation of the  $\text{SiO}_2$ .* The finely powdered silicate is heated before the blowpipe, with four times its weight of dry  $\text{Na}_2\text{CO}_3$ , till quite liquid. The melted mass is treated with  $\text{HCl}$ , evaporated to dryness without being filtered, and the residuc, after being moistened with concentrated  $\text{HCl}$ , (to change any insoluble oxichlorides which may have been formed during the evaporation, into soluble chlorides), and proceeded with as in sec. 82.

84. *b. Determination of the alkalis.*—An intimate mixture of one part of the silicate and five parts of  $\text{CaFl}_2$ , is stirred with  $\text{H}_2\text{SO}_4$ , till it becomes a thick paste. The mixture is at first gently warmed, and then heated more strongly till the excess of  $\text{H}_2\text{SO}_4$  has evaporated. The dry mass is boiled several times with fresh portions of water, and filtered; the  $\text{H}_2\text{SO}_4$  which is in solution, is removed by adding a solution of  $\text{BaCl}_2$ , and without filtering the fluid, lime-water is added, till an alkaline reaction is perceivable. The liquid is boiled;

the excess of CaO, and BaO precipitated with  $\text{NH}_3$  and  $(\text{NH}_4)_2\text{CO}_3$ , and treated further as in sec. 8, p. 40.

85.  $\text{HFl}$ . Silicates, which contain fluorides, evolve  $\text{SiFl}_4$ , when treated with concentrated sulphuric acid. If a glass rod, on the end of which a drop of water is hanging, be held close over the mixture, the water becomes covered with a film of  $\text{SiO}_2$ .—The silicate is fused with  $\text{Na}_2\text{CO}_3$ , as in sec. 83; but water is used to dissolve the fused mass. The aqueous solution, containing *all* the acids which may be present in the silicate ( $\text{Bo}_2\text{O}_3$ ,  $\text{HFl}$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ , etc.), is treated with  $\text{HCl}$ , till a distinctly acid reaction is apparent. An excess must be avoided, as  $\text{CaFl}_2$  is somewhat soluble in ammonia salts, especially in  $\text{NH}_4\text{Fl}$ . The liquid is now warmed to expel the  $\text{CO}_2$  which is dissolved, and  $\text{NH}_3$  is added till the reaction is alkaline. The precipitate of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , etc., is filtered off, and  $\text{CaCl}_2$  added to precipitate  $\text{CaFl}_2$ , which is confirmed as in sec. 64.— $\text{CaFl}_2$  is not decomposed by fusion with  $\text{Na}_2\text{CO}_3$ , unless  $\text{SiO}_2$  be present.

86. *Titanic acid*.— $\text{TiO}_2$  ( $\text{BaSO}_4$ ,  $\text{SrSO}_4$ ,  $\text{Al}_2\text{O}_3$ , etc.).  $\text{TiO}_2$ , which frequently accompanies silica, is found partly in the residue of  $\text{SiO}_2$ , and is partly precipitated with  $\text{Fe}_2\text{O}_3$ , and  $\text{Al}_2\text{O}_3$ .

*a.  $\text{TiO}_2$  and  $\text{SiO}_2$* .—The  $\text{SiO}_2$ , containing  $\text{TiO}_2$ , is boiled for some time with concentrated  $\text{H}_2\text{SO}_4$ , till some has evaporated. The mass is allowed to cool thoroughly, and poured, drop by drop, into an excess of  $\text{H}_2\text{O}$ , to avoid heating the fluid. It is then filtered, the most of the acid neutralised with  $\text{NaOH}$ , and the solution, which is still acid, boiled for some time. Precipitate:  $\text{TiO}_2$ .

Or they may be separated by the following method: The mixture of  $\text{SiO}_2$  and  $\text{TiO}_2$  is evaporated several times with  $\text{HFl}$  and  $\text{HCl}$  in a platinum crucible. If a residue remains, it consists of  $\text{TiO}_2$ , and such insoluble substances as  $\text{BaSO}_4$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{Cr}_2\text{O}_3$ , etc., which are not decomposable by fusion with  $\text{Na}_2\text{CO}_3$ . The residue is fused with  $\text{KHSO}_4$  in a platinum crucible, and the melted mass treated with cold water;  $\text{BaSO}_4$  and  $\text{SrSO}_4$

remain undissolved; the filtered solution is boiled for some time.  $\text{TiO}_2$  is precipitated, while  $\text{Al}_2\text{O}_3$ , and usually a trace of  $\text{TiO}_2$ , remains in solution.

b.  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ .—The precipitate of  $\text{Fe}_2\text{O}_3$ , containing  $\text{TiO}_2$ , is dissolved in dilute  $\text{H}_2\text{SO}_4$ , and nearly neutralised with  $\text{NaOH}$ ; a few drops of  $\text{HNO}_3$  are then added to the solution; it is diluted with water, and boiled for some time. Precipitate:  $\text{TiO}_2$ .

$\text{TiO}_2$ , heated in the oxidising flame of the blowpipe, with microcosmic salt, gives a yellow bead, which becomes colourless on cooling; in the reducing flame, a bead yellow when hot, which turns violet on cooling. The reaction is made more distinct if some tin foil be added to the bead. If Fe be present, the bead is red. It is usually difficult to detect  $\text{TiO}_2$ , by fusing the silicate alone with microcosmic salt.—An acid solution of  $\text{TiO}_2$  turns blue in presence of Zn; if ammonia be added, a blue precipitate of  $\text{Ti}_2\text{O}_3$  is thrown down.—If the precipitate of  $\text{TiO}_2$  be treated with Zn and  $\text{HCl}$ , it assumes a blue colour.

## ANALYSIS OF CYANIDES.

87. Of all the simple cyanides, only  $\text{HgCN}_2$  and the alkaline cyanides are soluble in  $\text{H}_2\text{O}$ . All cyanides, when warmed with concentrated  $\text{HCl}$ , give off free  $\text{HCN}$ ; it may be recognised by its smell. Hence the presence of cyanides may always be ascertained in dissolving the substance. Some of the double cyanides, e.g., potassic ferrocyanide, or ferricyanide, evolve only a part of their cyanogen as  $\text{HCN}$ . All cyanides, except those of the alkalis are decomposed on ignition.

88. Cyanogen is detected in the alkaline cyanides, as in XXXVII. 3, p. 32. In a mixture of ferrous and ferric salts, free  $\text{HCN}$  gives no blue precipitate. Potassic ferricyanide must first be formed by addition of excess of alkali. Cyanogen may be detected in  $\text{Hg}(\text{CN})_2$ , after the mercury is precipitated with  $\text{H}_2\text{S}$ .  $\text{AgNO}_3$  gives no

precipitate of  $\text{AgCN}$ , when added to an aqueous solution of  $\text{Hg}(\text{CN})_2$ .— $\text{AgCN}$  may be decomposed by ignition, and hence it differs from  $\text{AgCl}$ ,  $\text{AgBr}$ , and  $\text{AgI}$ . Cyanogen may be detected in other cyanides by distilling with  $\text{HCl}$ , adding excess of  $\text{NaOH}$  to the distillate, and proceed as in XXXVI. 3. Instead of the latter method, the dry cyanides may be fused with  $\text{Na}_2\text{CO}_3$  in a porcelain crucible, the melted mass dissolved in water, and the solution treated as directed above.

89. *Double cyanides of iron* may be easily decomposed by boiling with  $\text{NaOH}$ . The precipitate consists of the bases,  $\text{Fe}_2\text{O}_3$ , etc., which may be separated from each other in the usual manner; the solution contains  $4\text{KCN}$ ,  $\text{FeCN}_2$ ,  $3\text{KCy}$ ,  $\text{FeCN}_3$ , and the bases which are soluble in  $\text{NaOH}$  ( $\text{PbO}$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$ ). In testing for cyanogen in this solution, these oxides must be removed with  $\text{H}_2\text{S}$ . The solution is then acidified with  $\text{HCl}$ , and  $\text{FeCl}_3$  added to one portion of the solution, to test for  $4\text{KCN}$ ,  $\text{FeCN}_2$ ; while  $3\text{KCN}$ ,  $\text{FeCN}_3$  is tested for in another solution with  $\text{FeSO}_4$ . Turnbull's blue,  $3\text{FeCN}_2$ ,  $2\text{FeCN}_3$  (sec. 24), is decomposed, when boiled with  $\text{NaOH}$  into  $\text{Fe}(\text{OH})_2$  and potassic ferricyanide.

90. If it is required to establish the presence of these metals alone, which are in combination with cyanogen, the substance must be heated with a mixture of three parts of concentrated  $\text{H}_2\text{SO}_4$ , and one part of water, till the excess of  $\text{H}_2\text{SO}_4$  has volatilised. The residue is dissolved in  $\text{HCl}$ , and treated as usual. The alkalis which are present in a few insoluble cyanides, and which resist the solvent action of  $\text{H}_2\text{O}$  and of dilute acids, may also be detected in this manner.





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